Stability Report # 1836





**Analyses Report** 

Management System Information Laboratory

# Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20021134 Submission Id: 116305

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Time Label: 6 MONTHS Request Type: STABILITY Stability Study: 183G03

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

| Method        | Controls                          | Results     | Vers.        | M.U. | Spec? 1                  | ower lim                              | Upper lim | Spec? Lower lim Upper lim Book-Data Analyses | Analyst   | Verifier |
|---------------|-----------------------------------|-------------|--------------|------|--------------------------|---------------------------------------|-----------|--|-----------|----------|
| 083GCO02#00   | CAPSULE APPEARANCE                | 0=UNCHANGED | -            | ¥    |                          |                                       |           | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | CAPSULE COLOR                     | D=UNCHANGED | -            | æ    |                          |                                       |           | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | CONTENT APPEARANCE                | 0=UNCHANGED | -            | Ħ    |                          |                                       |           | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | CONTENT COLOR                     | 0≂UNCHANGED | <b></b>      | ¥    |                          |                                       |           | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | SMELL                             | 0=UNCHANGED | _            | æ    |                          |                                       |           | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
| 083GTI01#01_S | (HPLC TITER)<br>SAMPLE#1          | 97.95       | · <b>-</b>   | 86   |                          | - 000:06                              | - 110,000 | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | (HPLC TITER)<br>SAMPLE#2          | 97.96       | <b></b>      | ×    |                          | - 00006                               | - 110.000 | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | (HPLC TITER)<br>SAMPLE#3          | 97.25       | _            | %    |                          | - 000'06                              | - 110.000 | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | AVERAGE TITER (HPLC)              | 97.72       | -            | zę.  | ,                        | - 000'06                              | - 110.000 | 15545/18- MAY 2002                           | CAPPUCCIÓ | MARIAN   |
| 083GSC01#00   | TOTAL CORRELATED<br>SUBSTANCES    | 0.18        | -            | ×    | /                        | 21932                                 | ļ         | 16545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | IMPURITIES NOT LESS<br>THAN 0.05% | SEE TEXT    | -            | %    | COLON S                  | 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 | 18787     | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
| 083GKF01#00   | (HUMIDITY) SAMPLE #1              | 2,37        | <b>~</b>     | 3¢   | (81                      | pani                                  | 32        | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | (HUMIDITY) SAMPLE #2              | 2.4         | <del>-</del> | *    | हा <del>।</del><br>जिल्ल | i<br>Solo                             | 24        | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | (HUMIDITY) SAMPLE #3              | 2.38        | <b>-</b> -   | 8    | 18                       | Z.                                    | 29        | 15545/18- MAY 2002                           | CAPPUCCIO | MARIANI  |
|               | AVERAGE HUMIDITY                  | 2.20        |              | 3    | 13                       | Cillen                                | 3         | ***************************************      |           |          |

Printed



Analyses Report

Laboratory Information Management System

## Global Pharmaceutical Sciences - Italy

Submission Id: 116305 Sample ID (Request No.): 20021134 User sample ID (Iot): **I83G03**  Sample Type: SU010398 (PNU290940AD) ... CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183603
Pack Typer: 30 ml PLASTIC BOTTLE
Storage Condition: 25°CK50% U.R.
Time Label: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:
APPROVED
Status:
COMPLETE

| Samples Notes: |                               |         |          |            |       |                     |   |                    |           |          |
|----------------|-------------------------------|---------|----------|------------|-------|---------------------|---|--------------------|-----------|----------|
| Method         | Controls                      | Results | Vers.    | M.U.       | Spec? | Lower lim Upper lim | 1 | Book-Data Analyses | Analyst   | Verifier |
| 083GDS01#01    | SAMPLE#1                      | 94.01   | -        | ×          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | \$AMPLE#2                     | 99.04   | -        | %          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | SAMPLE#3                      | 100.16  | <b>*</b> | <b>Ж</b>   |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | SAMPLE#4                      | 94.01   | -        | *          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | SAMPLE#5                      | 88.41   | -        | *          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | SAMPLE#6                      | 94.58   | -        | ×          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | DISSOLUTION                   | 95.03   | -        | *          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | STANDARD DEVIATION            | 4.208   | -        | std-1      |       |                     |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | C.V.                          | 4.43    | -        | %          |       |                     |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | MIN. DISSOLUTION VALUE        | 88.41   | -        | %          |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|                | MAX. DISSOLUTION VALUE 100.16 | 100.16  | -        | <b>3</b> ₹ |       | 80.000              |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |

Printed



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.) ; 20021134 Submission Id: 116305

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290840AD) CAPSULE 25MG

Request Type: STABILITY

Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25^C/60% U.R. Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

condition: APPROVED

COMPLETE

Status:

Samples Notes:

Observations on the Method 083GC02#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 15 minutes: 76.5% (C.V.=13.9%)

Printed



Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Submission Id: 116305 Sample ID (Request No.); 20021134

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Preparation purpose: CLINICAL SUPPLY + STABILITY Request Type: STABILITY
Stability Study: 183G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25^C/60% U.R.
Time Label: 6 MONTHS

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 45 minutes: 99.97% (C.V.=1.16%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS than 0.05%

Total correlated substances; 0.18%

Printed



Analyses Report

Information Management Laboratory

System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20021134 User sample ID (lot): 183G03 Submission Id: 116305

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/60% U.R. Time Label: 8 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

5 / 10

Page



Analyses Report

Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Submission Id: 116305 Sample ID (Request No.): 20021134 User sample ID (lot): 183G03 Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25\*C/60% U.R. Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Area (%) RRT

svfbolla.rdf

20-MAY-2003 01:53 PM

Printed

by GIUSEPPE MACCARI



Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20021134 Submission Id: 116305

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG User sample ID (lot); 183G03

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Time Label: 6 MONTHS Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.06%

svíbolla.rdí



Analyses Report

Management System Information Laboratory

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20021134 Submission Id: 116305

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU280940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 mi PLASTIC BOTTLE Storage Condition: 25°C/60% U.R.

Time Label: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.12 99.0



Analyses Report

Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Submission id: 116305 Sample ID (Request No.); 20021134

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG-

Request Type: STABILITY
Stability Study: 183G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25\*C/60% U.R.
Time Label: 6 MONTHS
...Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.91

svíbolla.rdí

ģ 20-MAY-2003 01:53 PM

Printed

GIUSEPPE MACCARI

9 / 10

Page



Analyses Report

Laboratory Information Management

System

Global Pharmaceutical Sciences - Italy

Submission (d: 116305 Sample ID (Request No.); 20021134 User sample ID (lot): 183G03
Sample Type: SU010388 (PNU290840AD) CAPSULE
2SMG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/60% U.R.
Time Label: 8 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

1.00 99.82 (SU11248)

SUBMISSION APPROVED BY Volker ECK APPROVE DATE 15-may-2003

SAMPLE APPROVED BY: ROSARIA MARIANI APPROVE DATE: 17-may-2002

Printed

10 / 10

Page



Analyses Report

Laboratory Information Management System

### Global Pharmaceutical Sciences - Italy

Submission (d: 116307 Sample ID (Request No.); 20021136

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Samples Notes:

Request Type: STABILITY
Stability Study: I83G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40°C/75% U.R.
Time Labet: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:
APPROVED
Status:
COMPLETE

|               |                                   | Kesuits     | <u>i</u> |            | Spec | Lower IIm   | Lower lim Upper lim | Book-Data Analyses   | Analyst   | Verifier |
|---------------|-----------------------------------|-------------|----------|------------|------|---|---------------------|----------------------|-----------|----------|
| 083GCC02#00   | CAPSULE APPEARANCE                | 0=UNCHANGED | -        | ¥          |      |   |                     | 15645/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | CAPSULE COLOR                     | 0=UNCHANGED | -        | Ħ          |      |   |                     | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | CONTENT APPEARANCE                | 0=UNCHANGED | <b>~</b> | ¥          |      |   |                     | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | CONTENT COLOR                     | 0=UNCHANGED | -        | ¥          |      |   |                     | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | SMELL                             | 0=UNCHANGED | -        | Ħ          |      |   |                     | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
| 083GT101#01_S | (HPLC TITER)<br>SAMPLE#1          | 97.43       | -        | ઋ          |      | 90.000 - 110.000  | 110.000             | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | (HPLC TITER)<br>SAMPLE#2          | 98.55       | -        | %          |      | 90.000 - 110.000  | 110.000             | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | (HPLC TITER)<br>SAMPLE#3          | 97.23       | -        | %          |      | 90.000 - 110.000  | 110.000             | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | AVERAGE TITER (HPLC)              | 97.74       | -        | *          |      | 90.000 - 110.000  | 110,000             | 15545/18- MAY 2002   | CAPPUCCIO | MARIAN   |
| 083GSC01#00   | TOTAL CORRELATED SUBSTANCES       | 0.21        | *-       | *          | \    | 27.22.23  | /s                  | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | IMPURITIES NOT LESS<br>THAN 0.05% | SEE TEXT    | -        | 3¢         | E SI | 7955) (25 A)  | 25262               | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
| 083GKF01#00   | (HUMIDITY) SAMPLE #1              | 2.77        | -        | \$6        | (19  | Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve<br>Solve | 128                 | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | (HUMIDITY) SAMPLE #2:             | 2.9         | -        | <b>3</b> 8 | 151  | 000<br>0000   | 329                 | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |
|               | (HUMIDITY) SAMPLE #3              | 2.8         | -        | <b>%</b>   | 111  | (A)   | 30                  | / 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|               | AVERAGE HUMIDITY                  | 2.62        | ~        | <b>%</b>   | 217  |   | <u>जे</u> .         | 15545/18- MAY 2002   | CAPPUCCIO | MARIANI  |

Printed



Analyses Report

Management System Information Laboratory

Global Pharmaceutical Sciences - Italy

Submission Id: 116307 Sample ID (Request No.) : 20021136 User sample ID (lot) : **[83G03** 

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183603
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40^C/75% U.R.
Time Label: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

| Method<br>083GDS01#01 |                        |         |       |            |       |  |                    |            |          |
|-----------------------|------------------------|---------|-------|------------|-------|--|--------------------|------------|----------|
|                       | Controls               | Results | Vers. | Vers. M.U. | Spec? | Spec? Lower lim Upper lim Book-Data Analyses | Book-Data Analyses | Analyst    | Verifier |
|                       | SAMPLE#1               | 96.29   | -     | 3¢         |       | 80,000                                       | 15545/18- MAY 2002 | CAPPUCCIO  | MARIANI  |
|                       | SAMPLE#2               | 95.75   | -     | *          |       | 80.000                                       | 15545/18- MAY 2002 | CAPPUCCIO  | MARIAN   |
|                       | SAMPLE#3               | 96.82   | •     | 38         |       | 80.000                                       | 15545/18- MAY 2002 | CAPPUCCIO  | MARIANI  |
|                       | SAMPLE#4               | 100.05  | -     | 36         |       | 80.000                                       | 15545/16- MAY 2002 | CAPPUCCIO  | MARIANI  |
|                       | SAMPLE#5               | 99.51   | -     | 36         |       | 80.000                                       | 15545/18- MAY 2002 | CAPPUCCIO  | MARIAN   |
|                       | SAMPLE#6               | 97.90   | -     | 38         |       | 80.000                                       | 15545/18- MAY 2002 | CAPPUCCIO  | MARIAN   |
|                       | DISSOLUTION            | 97.72   | τ-    | <b>3</b> e |       | 80.000                                       | 15545/18- MAY 2002 | CAPPLICCIO | MARIANI  |
|                       | STANDARD DEVIATION     | 1.755   | -     | std-1      |       |  | 15545/18- MAY 2002 | CAPPUCCIO  | MARIAN   |
| _                     | c.v.                   | 8,1     | *~    | Ж.         |       |  | 15545/18- MAY 2002 | CAPPUCCIO  | MARIANI  |
| 7                     | MIN. DISSOLUTION VALUE | 95.75   | -     | %          |       | .000.08                                      | 15545/18- MAY 2002 | CAPPUCCIO  | MARIANI  |
| _                     | MAX. DISSOLUTION VALUE | 100,05  | -     | %          |       | 80.000                                       | 15545/18- MAY 2002 | CAPPUCCIO  | MARIANI  |

Printed



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.) : 20021135 Submission Id: 116307

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 40^C/75% U.R. Time Label: 8 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 15 minutes: 96.74% (C.V.= 1.77%)

Printed



Analyses Report

Management Information Laboratory

System

### Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20021136 User sample ID (lot): 183G03 Submission Id: 116307

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183603
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40^C/75% U.R.
Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE

#### Samples Notes:

Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 45 minutes: 97.72% (C.V.: 1.86%)

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances; 0,21%

Printed

GIUSEPPE MACCARI



Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Submission Id: 116307 Sample ID (Request No.): 20021136 User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY

Stability Study: 183603
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40°C/75% U.R.
Time Label: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%



Laboratory Information Management System

Global Pharmaceutical Sciences - Italy

Submission Id: 116307 Sample ID (Request No.): 20021136

User sample ID (lot): 183G03 Sample Type: SU010388 (PNU290940AD): CAPSULE 25MG

Request Type: STABILITY
Stability Study: I83G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40^C/75% U.R.
Time Label: 6 MONTHS

ilme Label: b MON1RS Preparation purpose: CLINICAL SUPPLY + STABILITY

Candition: APPROVED

Status: COMPLETE

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT Area (%)

Observations on the Method

svfbolla.rdf

20-MAY-2003 01:55 PM by GIUSEPPE MACCARI

Printed

Page 6 / 11



Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20021136 Submission ld: 116307

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Condition: APPROVED

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 40°C/75% U.R. Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

COMPLETE Status:

Samples Notes:

083GSC01#00, component IMPURITIES NOT LESS THAN 0.05%

Observations on the Method

7 / 11

Page



Analyses Report

Laboratory Information Management System

Global Pharmaceutical Sciences - Italy

Submission Id: 116307 Sample ID (Request No.): 20021136 User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study; 183603

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition; 40^C/75% U.R. Time Label: 6 MONTHS

Time Label: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.86 0.08

svfbolla.rdf

20-MAY-2003 01:55 PM by GIUSEPPE MACCARI

Printed

Page



**Analyses Report** 

Laboratory Information Management System

Global Pharmaceutical Sciences - Italy

Submission id: 116307 Sample ID (Request No.): 20021136 User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183503

Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 40ºC/75% U.R.
Time Label: 8 MONTHS
Preparation purpose: CLINICAL SUPPLY \* STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

0.91 0.06

svfbolla.rdf

20-MAY-2003 01:55 PM by GJUSEPPE MACCARI

Printed

Page



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20021138 Submission Id: 116307

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE: Storage Condition: 40°C/75% U.R. Time Label: 6 MONTHS

condition: APPROVED

COMPLETE Status:

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

0.07 0.94

svíbolia.rdf

20-MAY-2003 01:55 PM

Printed

by GIUSEPPE MACCARI

Page



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Submission Id: 116307

Sample ID (Request No.): 20021136 User sample ID (lot): 183G03 Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 40°C/75% U.R. Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

(SU11248) 99.79 9, SUBMISSION APPROVED BY Volker ECK
APPROVE DATE 15-may-2003

SAMPLE APPROVED BY; ROSARIA MARIANI APPROVE DATE: 17-may-2002

Printed



Analyses Report

Management System Information Laboratory

### Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20022083 Submission Id: 116953

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Pack Type: 30 mt PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Time Label: 9 MONTHS Request Type: STABILITY Stability Study: 183G03

condition: APPROVED Status: COMPLETE '

|            | i |
|------------|---|
| >          | Ì |
| <b>i</b> — | i |
| _          | í |
| ≡          |   |
| щ          | Į |
| ্ৰ         |   |
| - 55       |   |
| 0,         |   |
| +          | ŧ |
| >-         | i |
| _          |   |
| Д,         |   |
| п.         | į |
| $\supset$  |   |
| (C)        | į |
| _          | 1 |
| ⋖          |   |
| O          |   |
| =          |   |
| <b>=</b>   |   |
| پہ         |   |
| O          | į |
| • •        | 1 |
|            |   |
| Ä          |   |
| ŏ          |   |
| ┕          | į |
| =          | 3 |
| -          | ١ |
| 댿          | 1 |
| .0         |   |
| ₩          |   |
| 2          |   |
| æ          |   |
| -          |   |
| 2          |   |
| 0          |   |
|            |   |
|            |   |
|            |   |

| Samples Notes: |                                   |             |          |            |                |                              |            |                    |           |          |
|----------------|-----------------------------------|-------------|----------|------------|----------------|------------------------------|------------|--------------------|-----------|----------|
| Method         | Controls                          | Results     | Vers.    | M.U.       | Spec?          | Lower lim Upper lim          | aper lim   | Book-Data Analyses | Analyst   | Verifier |
| 083GCC002#00   | CAPSULE APPEARANCE                | 0=UNCHANGED | _        | ¥          |                |                              |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | CAPSULE COLOR                     | 0=UNCHANGED |          | B          |                |                              |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | CONTENT APPEARANCE                | 0=UNCHANGED |          | Ħ          |                |                              |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | CONTENT COLOR                     | 0=UNCHANGED | _        | 芸          |                |                              |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | SMELL                             | 0=UNCHANGED | _        | Ħ          |                |                              |            | 15545/33 AUG2002   | CAPPUCCIO | PINETTI  |
| 083GTI01#01_S  | (HPLC TITER)<br>SAMPLE#1          | 97.48       | ₩.       | <b>%</b>   |                | 90,000 - 11                  | - 110.000  | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | (HPLC TITER)<br>SAMPLE#2          | 96.12       | ~        | ×          |                | 90.000 - 11                  | - 110.000  | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | (HPLC TITER)<br>SAMPLE#3          | 97.03       | _        | *          |                | 90.000 - 110.000             |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | AVERAGE TITER (HPLC)              | 96.88       | -        | *          |                | 90.000 - 110.000             |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
| 083GSC01#00    | TOTAL CORRELATED SUBSTANCES       | 0.32        | -        | *          |                |                              |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | IMPURITIES NOT LESS<br>THAN 0.05% | SEE TEXT    | -        | <b>%</b>   | 1/3            | 40001222                     | /6         | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
| 083GKF01#00    | (HUMIDITY) SAMPLE #1              | 2.07        | -        | <b>3</b> 8 | S.             | <b>*</b>                     | (d)        | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | (HUMIDITY) SAMPLE #2              | 2.1         | _        | ×          | 19             | pane                         |            | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | (HUMIDITY) SAMPLE #3              | 2.2         | _        | *          | 工              | 500c                         | 777        | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                | AVERAGE HUMIDITY                  | 2.12        | <b>-</b> | »          | el <u>elli</u> | NAY SURAN<br>SURAN<br>A R CO | 82830      | 15545/33 AUG2002   | CAPPUCCIO | FINETTI  |
|                |                                   |             |          |            | 1.53           | 1234567                      | <b>8</b> 1 |                    |           |          |

Printed



Analyses Report

Management System Information Laboratory

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.) : 20022083 Submission Id: 116953

User sample ID (lot) : 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25-C/60% U.R. Time Label: 9 MONTHS Preparation purpose: CLINICAL SUPPLY + STABILITY Request Type: STABILITY Stability Study; 183G03

Condition: APPROVED

Status: COMPLETE

| Samples Notes: |                        |         |            |          |       |           |           |  |             |          |
|----------------|------------------------|---------|------------|----------|-------|-----------|-----------|--|-------------|----------|
| Method         | Controls               | Results | Vers.      | M.U.     | Spec? | Lower lim | Upper iim | Lower lim Upper lim Book-Data Analyses | Analyst     | Verifier |
| 083GDS01#01    | SAMPLE#1               | 89.91   | -          | ×        |       | 80.000    |           | 15545733 AUG2002                       | CAPPUCCIO   | FINETTI  |
|                | SAMPLE#2               | 88.29   | <b>-</b>   | <b>≫</b> |       | 80.000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETT   |
|                | SAMPLE#3               | 96.41   | <b>-</b> - | *        |       | 80,000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETT!  |
|                | SAMPLE#4               | 96.41   | <b></b>    | *        |       | 80.000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETT   |
| •              | SAMPLE#5               | 91.00   | -          | %        |       | 80.000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETTI  |
|                | SAMPLE#6               | 91.00   | _          | %        |       | 80,000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETT   |
|                | DISSOLUTION            | 92.17   | -          | æ        | -     | 80.000    |           | 16545/33 AUG2002                       | CAPPUCCIO   | FINETT   |
|                | STANDARD DEVIATION     | 3,431   | ***        | std-1    |       |           |           | 15545/33 AUG2002                       | CAPPUCCIO · | FINETTI  |
|                | C.V.                   | 3.72    | <b>-</b>   | %        |       |           |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETTI  |
|                | MIN. DISSOLUTION VALUE | 88.29   | -          | 36       |       | 80.000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETTI  |
|                | MAX. DISSOLUTION VALUE | 96.41   | -          | 26       |       | 80.000    |           | 15545/33 AUG2002                       | CAPPUCCIO   | FINETTI  |

Printed



Analyses Report

Laboratory Information Management

System

Global Pharmaceutical Sciences - Italy

Submission ld: 116953 Sample ID (Request No.): 20022083

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25^C/60% U.R. Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:
APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method

083GC002#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule



Analyses Report

Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20022083 Submission Id: 116953

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Time Label: 9 MONTHS

Condition: APPROVED

COMPLETE Status:

Samples Notes:

083GCO02#00, component: CAPSULE APPEARANCE Observations on the Method

containing yellow-orange powder

083GDS01#01, component: DISSOLUTION Observations on the Method

Average dissolution at 15 minutes: 74.93% (interval 63.91%-83.97%)

Printed



Laboratory Information Management System

Global Pharmaceutical Sciences - Italy

Submission Id: 116953 Sample ID (Request No.): 20022083

User sample ID (lot): **183G03**Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25^C/60% U.R.
Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method 083GDS01#01, component: DISSOLUTION

Average dissolution at 45 minutes: 96.56% (interval 95.33%-98.04%)

Observations on the Method 083GSC01#00, component; IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.32%



Management System Information Laboratory

Global Pharmaceutical Sciences - Italy

Submission Id: 116953 Sample ID (Request No.) 20022083 User sample ID (lot) 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Time Label: 9 MONTHS Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE

Samples Notes:

Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

svfbolla.rdf

6 / 11

Page



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy.

Sample ID (Request No.) 20022083 Submission Id; 116953

User sample ID (lot) [83G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25^C/60% U.R.
Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

AREA% RRT

svíbolta.rdf

Printed 20-MAY-2003 01:57 PM

by GIUSEPPE MACCARI

7 / 11

Page



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20022083 Submission Id: 116953

User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183G03
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25^C/60% U.R.
Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

99.0

svfbolla.rdf

Printed 20-MAY-2003 01:57 PM

by GIUSEPPE MACCARI

8 / 11 Page



Analyses Report

Management System Information

Laboratory

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.) 20022083 Submission id: 116953

User sample ID (lot) 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/50% U.R. Time Label: 9 MONTHS

Condition: APPROVED

Status: COMPLETE

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

0.05 0.94

svfbolla.rdf



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.) 20022083 Submission Id: 116953

User sample ID (lot) 183G03

Sample Type: SU010398 (PNU290840AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/60% U.R. Time Label: 9 MONTHS Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Stafus:

Samples Notes:

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

99.68 (SU11248) <del>6</del>

Observations on the Method

svfbolla.rdf

Printed



Analyses Report

Laboratory Information Management

System

Global Phaimaceutical Sciences - Italy

Submission Id: 116953 Sample ID (Request No.) 20022083

User sample ID (lot) [83G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183503

Pack Typo: 30 ml PLASTIC BOTTLE Storage Condition: 25^C/60% U.R. Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

condition: APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

1.06 0.16

SUBMISSION APPROVED BY VOKer ECK APPROVEDATE 15-may-2003

SAMPLE APPROVED BY: GIORGIO FINETTI APPROVE DATE: 03-sep-2002

Printed



Analyses Report

Management System Information Laboratory

### Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20022972 Submission Id: 117497

User sample ID (lot): 183G03

Samplo Typo: SU010398 (PNU290940AD) CAPSULE 25MG

Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25^C/60% U.R.
Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY Request Type: STABILITY Stability Study: 183G03

Condition: APPROVED Status: COMPLETE

| Samples Notes: |                                   |               |            |                              |                          |   |           |          |
|----------------|-----------------------------------|---------------|------------|------------------------------|--------------------------|---|-----------|----------|
| Method         | Controls                          | Results V     | Vers. M.U. | i, Spec? Lower lim Upper lim | r lim Book-Data Analyses |   | Analyst V | Verifier |
|                |                                   |               |            |                              |                          |   |           |          |
| 083GCO02#00    | CAPSULE APPEARANCE                | 0=UNCHANGED 1 | #          | ٠                            | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCARI  |
|                | CAPSULE COLOR                     | 0=UNCHANGED 1 | <b>\B</b>  |                              | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCARI  |
|                | CONTENT APPEARANCE                | 0=UNCHANGED 1 | <b>X</b>   |                              | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCARI  |
|                | CONTENT COLOR                     | 0≂UNCHANGED 1 | <b>x</b>   |                              | ,15545/69-NOV 2002       |   | CAPPUCCIO | MACCARI  |
| •              | SMELL                             | 0=UNCHANGED 1 | <b>5</b>   |                              | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCARI  |
| 083GTI01#01_S  | (HPLC TITER)<br>SAMPLE#1          | 95.84         | *          | 90.000 - 110.000             | .000 15545/59- NOV 2002  |   | CAPPUCCIO | MACCARI  |
|                | (HPLC TITER)<br>SAMPLE#2          | 96.17         | *          | 90.000 - 110.000             | .000 15545/59- NOV 2002  |   | CAPPUCCIO | MACCARI  |
|                | (HPLC TITER)<br>SAMPLE#3          | 97.03         | *          | 90.000 • 110.000             | .000 15545/59- NOV 2002  |   | CAPPUCCIO | MACCARI  |
|                | AVERAGE TITER (HPLC)              | 96.35         | *          | 90,000 - 110,000             | .000 15545/59- NOV 2002  |   | CAPPUCCIO | MACCARI  |
| 083GSC01#00    | TOTAL CORRELATED<br>SUBSTANCES    | 0.15          | *          | %<br>0002122232              | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCARI  |
|                | IMPURITIES NOT LESS<br>THAN 0.05% | SEE TEXT      | *          | Part Co.                     | 1554559- NOV 2002        |   | CAPPUCCIO | MACCARI  |
| 083GKF01#00    | (HUMIDITY) SAMPLE #1              | 2.1           | %          | Solo Contraction             | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCARI  |
|                | (HUMIDITY) SAMPLE #2              | 2.2           | *          | 2 2                          | 15545/59- NOV 2002       | • | CAPPUCCIO | MACCAR   |
|                | (HUMIDITY) SAMPLE #3              | 2.1           | *          | .63                          | 5 15545/59- NOV 2002     |   | CAPPUCCIO | MACCARI  |
|                | AVERAGE HUMIDITY                  | 2.13          | *          |                              | 15545/59- NOV 2002       |   | CAPPUCCIO | MACCAR   |
|                |                                   |               |            | 0                            |                          |   |           |          |

Printed



Analyses Report

Management Information Laboratory

System

# Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20022972 User sample ID (lot): 183G03 Submission Id: 117497

Sample Type: SU010398 (PNU290340AD) CAPSULE 25MG

Samples Notes:

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Request Type: STABILITY Stability Study: 183G03

Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED COMPLETE Status:

| and the second s |                        |          |       |          |       |          |         |  |           |          |
|--|------------------------|----------|-------|----------|-------|----------|---------|--|-----------|----------|
| Method   | Controls               | Results  | Vers. | M.U.     | Spec? |          | per lim | Lower lim Upper lim Book-Data Analyses | Analyst   | Verifier |
| 083GDS01#01  | SAMPLE#1               | 88,22    | τ-    | %        |       | 80.000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | SAMPLE#2               | 93.41    | -     | *        |       | 80.000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | SAMPLE#3               | 101.19   | -     | *        |       | 80.000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | SAMPLE#4               | 97.04    | -     | *        |       | . 000'08 |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | SAMPLE#5               | 94.97    | ₩.    | *        |       | 80.000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | SAMPLE#6               | 92.89    | •     | *        |       | 80,000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | DISSOLUTION            | 94,62    |       | *        |       | 90,000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCAR   |
|  | STANDARD DEVIATION     | 4.349    |       | std-1    |       |          |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | c.v.                   | 4.6      | +     | *        |       |          |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | MIN. DISSOLUTION VALUE | 88.22    | -     | *        |       | . 000'08 |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |
|  | MAX. DISSOLUTION VALUE | € 101.19 | -     | <b>%</b> |       | 80,000   |         | 15545/59- NOV 2002                     | CAPPUCCIO | MACCARI  |

Printed



. Analyses Report

Management System Information Laboratory

### Global Pharmaceutical Sciences - Italy

Sample ID (Request No.) ; 20022972 Submission Id: 117497

User sample ID (lot) .: 183G03

Sample Type: SU010398 (PNU290340AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/60% U.R. Time Label: 12 MONTHS

Condition: APPROVED Status: COMPLETE

|                | Sample 1996: Sucioses (FNOZSUSTARD) CAPSULE 25MG | Ozansavalj Ca | PSULE |      | Preparatio | Time Labei: 12 MUN LAS<br>Preparation purpose: CLINICAL SUPPLY + STABILITY | PPLY + STABILITY   |           |          |
|----------------|--|---------------|-------|------|------------|--|--------------------|-----------|----------|
| Samples Notes: | tes:   |               |       |      |            |  |                    |           |          |
| Method         | Controls   | Results       | Vers. | M.U. | Spec?      | Lower lim Upper lim  | Book-Data Analyses | Analyst   | Verifier |
| 083GMC01#      | 083GMC01#01TOTAL AEROBIC BACTERIA .1             | IN LIMITS     | -     | ž    |            | ABSENT   | 15545/59- NOV 2002 | CAPPUCCIO | MACCARI  |
|                |  |               |       |      |            | IN LIMITS  |                    |           |          |
|                |  |               |       |      |            | NOTIDETECTABLE   |                    |           |          |
|                |  |               |       |      |            | NOT PLANNED  |                    |           |          |
|                | ESCHERICHIA COLI                                 | ABSENT        | ٠-    | Ħ    |            | ABSENT   | 15545/59• NOV 2002 | CAPPUCCIO | MACCARI  |
|                |  |               |       |      |            | NOT DETECTABLE   |                    |           |          |
|                |  |               |       |      |            | NOT PLANNED  |                    |           |          |
|                | SALMONELLA                                       | ABSENT        | -     | ¥    |            | ABSENT   | 15545/59- NOV 2002 | CAPPUCCIO | MACCARI  |
|                |  |               |       |      |            | NOT DETECTABLE   |                    |           |          |
|                |  |               |       |      |            | NOT PLANNED  |                    |           |          |
|                | FUNGI  | INLIMITS      | -     | Ħ    |            | ABSENT   | 15545/59- NOV 2002 | CAPPUCCIO | MACCARI  |
|                |  |               |       |      |            | INLIMITS   |                    |           |          |
|                |  |               |       |      |            | NOT DETECTABLE   |                    |           |          |
|                | •  |               |       |      |            | NOT PLANNED  |                    |           |          |
|                | STAPHYLOCOCCUS AUREUS                            | ABSENT        | -     | ¥    |            | ABSENT   | 16545/59- NOV 2002 | CAPPUCCIO | MACCARI  |
|                |  |               |       |      |            | NOT DETECTABLE   |                    |           | •        |
|                |  |               |       |      |            | NOT PLANNED  |                    |           |          |
|                | ENTEROBACTERIA                                   | IN LIMITS     | •     | 茎    |            | ABSENT   | 15545/59- NOV 2002 | CAPPUCCIO | MACCARI  |
|                |  |               |       |      |            | IN LIMITS  |                    |           |          |
|                |  |               |       |      |            | NOT DETECTABLE   |                    |           |          |
|                |  |               |       |      |            |  |                    |           |          |

Printed



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Submission Id: 117497 Sample ID (Request No.); 20022972 User sample ID (lot): 183G03 Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25/C/60% U.R.
Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

condition: APPROVED Status: COMPLETE

| Samples Notes:  | tes:                       |          |       |      |               |             |          |   |         |          |
|-----------------|----------------------------|----------|-------|------|---------------|-------------|----------|---|---------|----------|
| Method Controls | Method Controls Results    | 1        | Vers. | M.U. | M.U. Spec? Lo | Lower lim   | Upperlim | Vers. M.U. Spec? Lowerlim Upperlim Book-Data Analyses | Analyst | Verifier |
| 3GMC01#01       | 083GMC01#01 ENTEROBACTERIA | INLIMITS | -     | B    |               | NOT PLANNED |          |   |         |          |



Analyses Report

Laboratory Information Management

System

Global Pharmaceutical Sciences - Italy

Submission Id: 117497 Sample ID (Request No.): 20022972

User sample ID (lot); **183G03** 

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/80% U.R.

Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:
APPROVED

Status: COMPLETE

Samples Notes:

Observations on the Method 083GC002#00, component: CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDSD1#01, component: DISSOLUTION

Average dissolution at 15 minutes: 77.24% (C.V.: 7.27%)

Printed



Analyses Report

Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20022972 Submission Id: 117497

User sample ID (lot): [83G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/60% U.R. Time Label: 12 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED COMPLETE

Samples Notes:

Observations on the Method 083GDS01#01, component: DISSOLUTION

Average dissolution 45 minutes: 97.39% (C.V.: 2.25%)

083GMC01#01, component: TOTAL AEROBIC BACTERIA Observations on the Method

Total bacterial count < 10 Utc/g (Report num, 02.16971)

Observations on the Method 083GMC01#01, component: ENTEROBACTERIA

Enterobacteria < 10 Ufc/g (Report num. 02.16971)

083GMC01#01, component: ESCHERICHIA COLI

Observations on the Method

Report num. 02.16971

083GMC01#01, component: FUNGI Observations on the Method

Fungi (Yeasts + Molds) < 10 Ufc/g (Report num. 02.16971)

083GMC01#01, component: SALMONELLA Observations on the Method

Report num. 02.16971

083GMC01#01, component: STAPHYLOCOCCUS AUREUS Observations on the Method

Report num. 02.16971

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

Total correlated substances: 0.15%



Analyses Report

Management Information Laboratory

System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.); 20022972 User sample ID (lot): 183G03 Submission Id; 117497

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/80% U.R.
Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

svfbolla.rdf

Page

by GIUSEPPE MACCARI

20-MAY-2003 01:56 PM

Printed



Analyses Report

Information Management Laboratory System

Global Pharmaceutical Sciences - Italy

Submission Id: 117497 Sample ID (Request No.) 20022972 User sample ID (lot) 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY
Stability Study: 183603
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25AC/60% U.R.
Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GC01#00, component: IMPURITIES NOT LESS THAN 0.05%

RRT

Area (%)

Printed



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Submission Id: 117497 Sample ID (Request No.); 20022972 User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25°C/60% U.R.
Time Labei: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

Observations on the Method 083GC01#00, component: IMPURITIES NOT LESS THAN 0.05%



Analyses Report

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Submission Id: 117497 Sample ID (Request No.) 20022972 User sample ID (lot) 183G03 Sample Type: SU010398 (PNU290840AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storago Condition: 25/C/60% U.R. Time Label: 12 MONTHS Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

083GC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

0.10 0.65



**Analyses Report** 

Management Information Laboratory System

Global Pharmaceutical Sciences - Italy

Submission (d: 117497 Sample ID (Request No.) 20022972

User sample ID (lot) 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Request Type: STABILITY Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25^C/60% U.R. Time Label: 12 MONTHS Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Samples Notes:

083GC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

0.05 9.9



#### Analyses Report

Management information Laboratory System

Global Pharmaceutical Sciences - Italy

Sample ID (Request No.): 20022972 Submission Id: 117497

User sample ID (lot): 183G03

Sample Type: SU010338 (PNU290340AD) CAPSULE 25MG.

Request Type: STABILITY
Stability Study: 83503
Pack Type: 30 ml PLASTIC BOTTLE
Storage Condition: 25/C/80% U.R.
Time Label: 12 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED COMPLETE Status:

Samples Notes:

083GC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

99.85 (SU 11248) 1.00 SUBMISSION APPROVED BY Volker ECK APPROVE DATE 15-may-2003

SAMPLE APPROVED BY; GIUSEPPE MACCARI APPROVE DATE: 27-nov-2002

svfbolla.rdf

Time Points



Integrated Analytical Bulletin

Management System Laboratory Information

#### Pharmaceutical Development

Sample ID (Request No.): 20022083 Submission Id: 116953

User sample ID (lot): 183G03

Sample Date: 18/07/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Stability Study Start Date: 18/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano Samples Notes:

Time Label: 9 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25AC/60% U.R. Stability Study: 183603

Batch Manufacture Date: 17/09/2001

Condition: APPROVED

Status:

|   | ш                |
|---|------------------|
|   | 面                |
|   | ☱                |
| • | 으                |
| 3 | ≥                |
|   | Х                |
| , | $\mathbf{\circ}$ |

|             | Comple                            |             | vers. m.o. | Spec? Lowe   | Lower lim Upper lim | Book-Data Analyses | Analyst   | Verifier |
|-------------|-----------------------------------|-------------|------------|--------------|---------------------|--------------------|-----------|----------|
| 0836CO02#0  | CAPSULE APPEARANCE                | 0=UNCHANGED | ጃ          |              |                     | 15545/32 AUG2002   | CAPPUCCIO | FINET    |
|             | CAPSULE COLOR                     | 0=UNCHANGED | ጀ          |              |                     | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
|             | CONTENT APPEARANCE                | 0=UNCHANGED | ¥          |              |                     | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
|             | CONTENT COLOR                     | 0=UNCHANGED | ¥          |              |                     | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
|             | SMELL                             | 0=UNCHANGED | ጀ          |              |                     | 15545/32 AUG2002   | CAPPUCCIO | FINETT   |
| 08367101#01 | (HPLC TITER) SAMPLE#1             | 97.48       | %          | 90.000       | 00 - 110.000        | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
| o,          | (HPLC TITER) SAMPLE#2             | 96,12       | %          | 90.000       | 00 - 110.000        | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
|             | (HPLC TITER) SAMPLE#3             | 97.03       | *          | 90.000       | 00 - 110.000        | 15545/32 AUG2002   | CAPPUCCIO | FINETT   |
|             | AVERAGE TITER (HPLC)              | 98,88       | %          | 90.000       | 00 - 110.000        | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
| 083GSC01#0  | TOTAL CORRELATED                  | 0,32        | 8          |              |                     | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
|             | IMPURITIES NOT LESS THAN<br>0.05% | SEE TEXT    | %          | THE STATE OF | (S                  | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
| 083GKF01#00 | (HUMIDITY) SAMPLE #1              | 2.07        | %          | A Project    |                     | 15545/32 AUG2002   | CAPPUCCIO | FINETTI  |
|             | (HUMIDITY) SAMPLE #2              | 2.1         | %          | W The New W  | 23<br>Q             | 15545/32 AUG2002   | CAPPUCCIO | FINE     |
|             | (HUMIDITY) SAMPLE #3              | 2.2         | %          | 2005 M       | 44                  | 15545/32 AUG2002 · | CAPPUCCIO | FINETT   |
|             | AVERAGE HUMIDITY                  | 2.12        | %          | 20           | 6;                  | 15545/32 AUG2002   | CAPPUCCIO | FINETT   |

syfbollaintegrated.rdf

Printed 22-AUG-2002 10:15 AM

MACCARI

Page



Integrated Analytical Bulletin

Management System Laboratory Information

Condition: APPROVED

Status: COMPLETE

#### Pharmaceutical Development

Sample ID (Request No.): 20022083 Submission ld: 116953

User sample ID (lot): 183G03

Sample Date: 18/07/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Preparation purpose: CLINICAL SUPPLY + STABILITY

Pack Type: 30 ml PLASTIC BOTTLE

Batch Manufacture Date: 17/09/2001 Stability Study: 183G03 Storage Condition: 25^C/60% U.R.

Time Label: 9 MONTHS

Stability Study Start Date: 16/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano

Samples Notes:

| Wethod     | Controls               | Results | Vers. | M.U.       | Spect |        | Upper lim | Lower lim Upper lim Book-Data Analyses | Analyst   | Verifier |
|------------|------------------------|---------|-------|------------|-------|--------|-----------|--|-----------|----------|
| 083GDS01#0 | SAMPLE#1               | 89.91   |       | · %        |       | 80.000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETTI  |
|            | SAMPLE#2               | 88.29   |       | *          |       | 80.000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETTI  |
|            | SAMPLE#3               | 96.41   |       | <b>%</b> € |       | 80.000 |           | 15545/32, AUG2002                      | CAPPUCCIO | FINETTI  |
|            | SAMPLE#4               | 96,41   |       | %          |       | 80.000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETTI  |
|            | SAMPLE#5               | 91.00   |       | 96         |       | 80.000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETTI  |
|            | SAMPLE#6               | 91.00   |       | %          |       | 80.000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETT   |
|            | DISSOLUTION            | 92.17   |       | *          |       | 80.000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETTI  |
|            | STANDARD DEVIATION     | 3.431   |       | std-1      |       |        |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETTI  |
|            | C.V.                   | . 3.72  |       | *          |       |        |           | 15545/32 AUG2002                       | CAPPUCCIO | FINETT   |
|            | MIN. DISSOLUTION VALUE | 88.29   |       | %          |       | 80,000 |           | 15545/32 AUG2002                       | CAPPUCCIO | FIRET    |
|            | MAX. DISSOLUTION VALUE | 96.41   |       | %          |       | 80,000 |           | 15545/32 AUG2002                       | CAPPUCCIO | PINETTI  |

Printed



Integrated Analytical Bulletin

Management System Information Laboratory

#### Pharmaceutical Development

Sample ID (Request No.): 20022083 Submission Id: 116953

User sample ID (lot): [83G03

Sample Date: 18/07/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Stability Study Start Date: 18/10/2001
Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmadia Nerviano

Batch Manufacture Date: 17/09/2001

Pack Type: 30 ml PLASTIC BOTTLE Stability Study: 183G03

Storage Condition: 25/C/60% U.R. Time Label: 9 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE Status:

Samples Notes:

083GC002#00, component: CAPSULE APPEARANCE Observations on the Method Opaque brick red colored, format 3, hard gelatin capsule containing red-orange powder

083GDS01#01, component: DiSSOLUTION Observations on the Method

Average dissolution at 15 minutes; 74.93% (interval 63.91%-89.37%)

Average dissolution at 45 minutes: 96.59% (interval 95.33%-98.04%)

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

Total correlated substances; 0,32%

0.05 99.68 (SU11248) 0.16

0.66 0.94 1.00 1.06

05-aug-2002 Approved by: FINETTI Approval date:

syfbollaintegrated.rdf

22-AUG-2002 10:15 AM

Printed

by MACCARI

3 \_ 3



Management System Information Laboratory

# Integrated Analytical Bulletin

#### Pharmaceutical Development

Sample ID (Request No.): 20021134 User sample ID (lot): 183G03 Submission Id: 116305

Sample Date: 18/04/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Batch Manufacture Date: 17/09/2001 Stability Study: 183G03

Time Label: 6 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25AC/60% U.R.

Condition: APPROVED Status: COMPLETE

|  | Nerviano  |
|--|---|
|  | tability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano |
| -                                      | acturer: PE   |
| Stability Study Start Date: 18/10/2001 | 017 - Manuf   |
| tart Date:                             | pher: 20010   |
| Study S                                | otocol Nun  |
| Stability                              | tability Pr   |

| Method      | Controls                         | Results     | M.U. | Spec? Low     | Spec? Lower lim Upper lim                 | Book-Data Analyses | Analyst   | Verifier |
|-------------|----------------------------------|-------------|------|---------------|---|--------------------|-----------|----------|
| 083GCO02#0  | CAPSULE APPEARANCE               | D=UNCHANGED | Ħ    |               | <del></del>                               | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | CAPSULE COLOR                    | 0=UNCHANGED | Ħ    |               | •   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | CONTENT APPEARANCE               | 0=UNCHANGED | ፰    | •             | ,-  | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | CONTENT COLOR                    | 0=UNCHANGED | Ħ    |               | •   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | SMELL                            | 0=UNCHANGED | Ħ    |               |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 083GTI01#01 | (HPLC TITER) SAMPLE#1            | 95'26       | 32   | 90.000        | 90.000 - 110.000                          | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| တ္          | (HPLC TITER) SAMPLE#2            | 97.98       | %    | 90.000        | - 110.000                                 | 15545/18- MAY 2002 | CAPPUCCIO | MARIAN   |
|             | (HPLC TITER) SAMPLE#3            | 97.25       | *    | 30.000        | 90.000 - 110.000                          | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | AVERAGE TITER (HPLC)             | 97.72       | *    | 90.000        | - 110.000                                 | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 083GSC01#0  | TOTAL CORRELATED SUBSTANCES 0.18 | , 0.18.     | %    |               |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | IMPURITIES NOT LESS THAN 0.05%   | SEE TEXT    | %    | 10000         |   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 083GKF01#00 | (HUMIDITY) SAMPLE #1             | 2.37        | *    | TO THE PARTY. | /\$                                       | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | (HIMIDITY) SAMPIF#2              | 2.4         | %    | di<br>di      | 23  | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             | (HUMIDITY) SAMPLE #3             | 2.38        | %    | a November    | ¥51                                       | 15545/18- MAY 2002 | CAPPUCCIO | MARIAN   |
|             | AVERAGE HUMIDITY                 | 2.38        | *    | DOZ MAN       | 378<br>N                                  | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
|             |                                  |             |      | 37077<br>1077 | 9 1077                                    |                    |           |          |
|             |                                  |             |      | 415767776     | \$\$ \$\\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ |                    |           |          |

Page

by MARIANI

Printed 17-MAY-2002 05:05 PM

svíboliaintegrated.rdf



### Integrated Analytical Bulletin

Management System Information Laboratory

#### Pharmaceutical Development

Submission Id: 116305

Sample ID (Request'No.): 20021134 User sample ID (lot): **§83G03** 

Sample Date: 18/04/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Stability Study Start Date: 18/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano

Samples Notes

Batch Manufacture Date: 17/09/2001 Stability Study: 183G03 Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R.

Time Label: 6 MONTHS

Condition: APPROVED Status: COMPLETE

| Ę        |
|----------|
| 퍨        |
| STA      |
| ŧ        |
| Ť        |
| S        |
|          |
| ₹        |
| CLINICAL |
|          |
| pos      |
| Ε        |
| -<br>-   |
| raţi     |
| ede      |
| ڇ        |

| -          | And the second second control of the second sec | Married Control of the Party of | The state of the last of the l | STATE OF THE PERSONS | -         | Section of the least of the lea |  | STATE OF THE PERSON OF THE PER | The second second |
|------------|--|--|--|----------------------|-----------|--|--|--|-------------------|
| Method     | Controls   | Results  | M.U.   | Spec?                | Lower lim | Upper lim  | Lower lim Upper lim Book-Data Analyses | Analyst  | Verifier          |
| 083GDS01#0 | SAMPLE#1   | 94.01  | %  |                      | 80,000    |  | 1554S/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | SAMPLE#2   | 99.04  | %  |                      | 80,000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | SAMPLE#3   | 100.16   | %  |                      | 80.000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | SAMPLE#4   | 94.01  | %  |                      | 80.000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | SAMPLE#5   | 88.41  | *  |                      | 80.000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | SAMPLE#6   | 94.56  | %  |                      | 80,000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | DISSOLUTION  | 95.03  | %  |                      | 80.000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | STANDARD DEVIATION   | 4.208  | . std-1  |                      |           |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | C,V.   | 4.43   | <b>%</b>   |                      |           |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | MIN. DISSOLUTION VALUE   | 88.41  | æ  |                      | 80.000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIANI           |
|            | MAX. DISSOLUTION VALUE   | 100.16   | *  |                      | 80.000    |  | 15545/18- MAY 2002                     | CAPPUCCIO  | MARIAN            |



### Integrated Analytical Bulletin

Management Information Laboratory System

Condition: APPROVED

Status: COMPLETE

#### Pharmaceutical Development

Sample ID (Request No.) 20021134 Submission Id: 116305

User sample ID (lot) 183G03

Sample Date: 18/04/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Preparation purpose: CLINICAL SUPPLY + STABILITY

Pack Type: 30 ml PLASTIC BOTTLE

Batch Manufacture Date: 17/09/2001 Stability Study: 183G03 Storage Condition: 25^C/60% U.R. Time Label; 6 MONTHS

Stability Study Start Date: 16/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano

Samples Notes:

Observations on the Method 083GC002#00, component; CAPSULE APPEARANCE

Opaque brick red colored, format 3, hard gelatin capsule containing red-orange powder

083GDS01#01, component: DISSOLUTION Observations on the Method

Dissolution at 15 minutes: 76.5 % (C.V.≈ 13.9%)
Dissolution at 45 minutes: 99.97% (C.V.≖ 1.16%)

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

Total correlated substances: 0.18%

Area (%) ART

0.12 0.66

90.0

99.82 (SU11248) 1.00

Approved by: MARIANI

17-may-2002

Approval date:

Printed

svfbollainfegrafed.rdf

17-MAY-2002 05:05 PM

3/3

by MARIANI



Integrated Analytical Bulletin

Management System Information Laboratory

#### Pharmaceutical Development

Sample ID (Request No.): 20021136 Submission Id: 116307

User sample ID (lot): 183G03

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano Stability Study Start Date: 18/10/2001

Preparation purpose: CLINICAL SUPPLY + STABILITY Storage Condition; 40^C/75% U.R. Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG Sample Date: 18/04/2002

Condition: APPROVED

Pack Type: 30 ml PLASTIC BOTTLE

Batch Manufacture Date: 17/09/2001 Stability Study: 183G03 Time Label: 6 MONTHS

Status: COMPLETE

| CAPPLICATION   CAPPLIE APPEARANCE   CHUNCHANGED   DIT  | Method         | Controls                       | Results     | M.U. | Spec?                 | Lower IIm                 | Lower Itm Upper Itm | Book-Data Analyses | Analyst   | Verifier |
|--|----------------|--------------------------------|-------------|------|-----------------------|---------------------------|---------------------|--------------------|-----------|----------|
| CONTENT APPEARANCE         LUNCHANGED         txt         15545/18- MAY 2002         CAPPUCCIO           CONTENT APPEARANCE         0=UNCHANGED         txt         15545/18- MAY 2002         CAPPUCCIO           CONTENT COLOR         0=UNCHANGED         txt         15545/18- MAY 2002         CAPPUCCIO           SMELL         0=UNCHANGED         txt         90,000         110,000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#2         98.55         %         90,000         110,000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#3         97.23         %         90,000         110,000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90,000         110,000         15545/18- MAY 2002         CAPPUCCIO           TOTAL CORRELATED SUBSTANCES         2.21         %         90,000         110,000         15545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#2         2.37         %         90,000         110,000         15545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#2         2.82         %         15545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#2         2.82         %         15545/18- MAY 2002 <td>083GCO02#0</td> <td>CAPSULE APPEARANCE</td> <td>0=UNCHANGED</td> <td>¥</td> <td></td> <td></td> <td></td> <td>15545/18- MAY 2002</td> <td>CAPPUCCIO</td> <td>MARIANI</td>   | 083GCO02#0     | CAPSULE APPEARANCE             | 0=UNCHANGED | ¥    |                       |                           |                     | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| CONTENT APPEARANCE         0=UNCHANGED         bt         15545/18- MAY 2002         CAPPUCCIO           CONTENT COLOR         0=UNCHANGED         bt         15545/18- MAY 2002         CAPPUCCIO           SMELL         0=UNCHANGED         bt         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#1         97.43         %         90.000         110.000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#2         98.55         %         90.000         110.000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#3         97.74         %         90.000         110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         110.000         15545/18- MAY 2002         CAPPUCCIO           MPURITIES NOT LESS THAN 0.05%         SEE TEXT         %         AVERAGE         CAPPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.82         %         AVERAGE         CAPPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.82         %         AVERAGE         CAPPUCCIO </td <td>0</td> <td>CAPSULE COLOR</td> <td>0=UNCHANGED</td> <td>¥</td> <td></td> <td></td> <td></td> <td>15545/18- MAY 2002</td> <td>CAPPUCCIO</td> <td>MARIANI</td>   | 0              | CAPSULE COLOR                  | 0=UNCHANGED | ¥    |                       |                           |                     | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| CONTENT COLOR         0=UNCHANGED         txt         15545/18-MAY 2002         CAPPUCCIO           SMELL         0=UNCHANGED         txt         15545/18-MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#1         97.43         %         90.000         110.000         15545/18-MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#2         98.55         %         90.000         -110.000         15545/18-MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         -110.000         15545/18-MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         -110.000         15545/18-MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         -110.000         15545/18-MAY 2002         CAPPUCCIO           IMPURITIES NOT LESS THAN 0.05%         SEE TEXT         %         AVERAGE MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #1         2.77         %         AVERAGE MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.82         %         AVERAGE MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.82         %         AVERAGE MAY 2002         CAPPUCCIO           (  |                | CONTENT APPEARANCE             | 0=UNCHANGED | ž    |                       |                           |                     | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| SMELL         deunchanged         bx         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#1         97.43         %         90.000 · 110.000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#2         98.55         %         90.000 · 110.000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#3         97.74         %         90.000 · 110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000 · 110.000         15545/18- MAY 2002         CAPPUCCIO           INFULZITIES NOT LESS THAN 0.05%         SEE TEXT         %         15545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#1         2.77         %         AND  |                | CONTENT COLOR                  | 0=UNCHANGED | ¥    |                       |                           |                     | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| (HPLC TITER) SAMPLE#1         97.43         %         90.0000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#2         98.55         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           (HPLC TITER) SAMPLE#3         97.23         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           TOTAL CORRELATED SUBSTANCES         0.21         %         15545/18- MAY 2002         CAPPUCCIO           IMPURITIES NOT LESS THAN 0.05%         SEE TEXT         %         77         %         7545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #2         2.9         %         77         %         7545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.8         %         7545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.8         %         7545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.8         %         7545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.8         %         7545/18- MAY 2002 <t< td=""><td></td><td>SMELL</td><td>0=UNCHANGED</td><td>Ħ</td><td></td><td></td><td></td><td>15545/18- MAY 2002</td><td>CAPPUCCIO</td><td>MARIANI</td></t<>  |                | SMELL                          | 0=UNCHANGED | Ħ    |                       |                           |                     | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| (HPLC TITER) SAMPLE#2         98.55         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.23         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           TOTAL CORRELATED SUBSTANCES         0.21         %         15545/18- MAY 2002         CAPPUCCIO           IMPURITIES NOT LESS THAN 0.05%         SEE TEXT         %         15545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #1         2.77         %         15545/18- MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #2         2.8         %         MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.82         %         MAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.82         %         MAY 2002         CAPPUCCIO <td>083GT101#01</td> <td>(HPLC TITER) SAMPLE#1</td> <td>97.43</td> <td>%</td> <td></td> <td>90,000</td> <td>- 110,000</td> <td>15545/18- MAY 2002</td> <td>CAPPUCCIO</td> <td>MARIANI</td>   | 083GT101#01    | (HPLC TITER) SAMPLE#1          | 97.43       | %    |                       | 90,000                    | - 110,000           | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| (HPLC TITER) SAMPLE#3         97.23         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           AVERAGE TITER (HPLC)         97.74         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           TOTAL CORRELATED SUBSTANCES         0.21         %         CAPPUCCIO         CAPPUCCIO         CAPPUCCIO           IMPURITIES NOT LESS THAN 0.05%         SETEXT         %         TAPPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE#1         2.77         %         ANAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#3         2.8         %         ANAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#3         2.8         %         ANAY 2002         CAPPUCCIO           (HUMIDITY) SAMPLE#3         2.82         %         ANAY 2002         CAPPUCCIO   | ν <sub>ι</sub> | (HPLC TITER) SAMPLE#2          | 98.55       | %    |                       |                           | - 110,000           | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| AVERAGE TITER (HPLC)         97.74         %         90.000         - 110.000         15545/18- MAY 2002         CAPPUCCIO           TOTAL CORRELATED SUBSTANCES         0.21         %         15545/18- MAY 2002         CAPPUCCIO           IMPURITIES NOT LESS THAN 0.05%         SEE TEXT         %         TAPPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE #1         2.77         %         APPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE #2         2.8         %         APPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.8         %         APPUCCIO         CAPPUCCIO           (HUMIDITY) SAMPLE #3         2.8         %         APPUCCIO         CAPPUCCIO           AVERAGE HUMIDITY         2.82         %         APPUCCIO         CAPPUCCIO           AVERAGE HUMIDITY         2.82         %         APPUCCIO         CAPPUCCIO  |                | (HPLC TITER) SAMPLE#3          | 97.23       | *    |                       | 90.000                    | - 110.000           | 15545/18- MAY 2002 | CAPPUCCIO | MARIAN   |
| 15545/18- MAY 2002   15545/1   |                | AVERAGE TITER (HPLC)           | 97.74       | *    |                       | 90,000                    | - 110.000           | 15545/18- MAY 2002 | CAPPUCCIO | MARIAN   |
| THAN 0.05% SEE TEXT % 15545/18- MAY 2002 CAPPUCCIO 2.9 % 15545/18- MAY 2002 CAPPUCCIO 2.9 % 15545/18- MAY 2002 CAPPUCCIO 2.8 % 15545/18- MAY 2002 CAPPUCCIO 2.8 % 15545/18- MAY 2002 CAPPUCCIO 2.82 % 15545/18- MAY 2002 CAPPUCCIO 3.82 % 15545/18- MAY 2002 CAPPUCCIO 3.84 % 15545/18- MAY 2002 CAPPUCCIO   | 083GSC01#0     | TOTAL CORRELATED SUBSTANCES    | 0.21        | *    |                       |                           |                     | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 2.8 % A MAY 2002 CAPPUCCIO 2.8 % A MAY 2002 CAPPUCCIO 2.8 % A MAY 2002 CAPPUCCIO 3.82 % A MAY 2002 CAPPUCCIO 5.82 % A MAY 2002 CAPPUCCIO 5.82 % A MAY 2002 CAPPUCCIO 5.82 % A MAY 2002 CAPPUCCIO   |                | IMPURITIES NOT LESS THAN 0.05% | SEE TEXT    | 36   | /                     | 29303                     | ,                   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 2.8 % MAY 2002 CAPPUCCIO CAPPUCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CA | 083GKF01#00    | (HUMIDITY) SAMPLE#1            | 2.77        | *    | Ede                   | 4                         | <u> </u>            | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 2.82 % (A MAY 2002 CAPPUCCIO 15545/18- MAY 2002 CAPPUCCIO Sugal  |                | (HUMIDITY) SAMPLE #2           | 2.9         | %    | `<br>\$≥ <sub>1</sub> | , OC. 1                   | _                   | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 2.82 % NAM 2002 CAPPUCCIO SUBOR O CAPPUCCIO A B CO CAPPUCCIO CAPPUCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCCIO CAPPUCI |                | (HUMIDITY) SAMPLE #3           | 2.8         | %    | Se de                 | <b>200</b>                | 56                  | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| CO MANY CONTRACTOR OF THE PARTY |                | AVERAGE HUMIDITY               | 2.82        | %    | - T                   | 1<br>70<br>72<br>73<br>73 | 78                  | 15545/18- MAY 2002 | CAPPUCCIO | MARIANI  |
| 12 13 14 15 15 15 16 16 16 16 16 16 16 16 16 16 16 16 16   |                |                                |             |      | 7120th                | SUBOIL<br>A & CD          | 270/                |                    |           |          |
|  |                |                                |             |      |                       | 3141010                   | ઢે                  |                    |           |          |

Printed



Integrated Analytical Bulletin

Management System Inforrnation Laboratory

#### Pharmaceutical Development

Sample (D (Request No.): 20021136 User sample ID (lot): 183G03 Submission Id: 116307

Sample Type: SU010398 (PNU290940AD) CAP 25MG Sample Date: 18/04/2002

Stability Study Start Date: 18/10/2001 Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmada Nerviano

Pack Type: 30 ml PLASTIC BOTTLE Batch Manufacture Date: 17/09/2001 Stability Study: 183G03

Storage Condition: 40vC/75% U.R. Time Label: 6 MONTHS

Condition: APPROVED COMPLETE Status:

STABILITY

| +        |  |
|----------|--|
| չ        |  |
|          |  |
| SUPP     |  |
|          |  |
| #ICAL    |  |
| ž        |  |
| Š        |  |
| 0        |  |
| se       |  |
| 6        |  |
| Ĭ        |  |
| <u>-</u> |  |
| -23      |  |
| 20       |  |
| eba      |  |
| ĕ        |  |
| _        |  |
|          |  |
|          |  |
|          |  |
|          |  |
|          |  |
| Щ        |  |
| 5        |  |
| يَد      |  |

| Method     | Controls               | Results | M.U.      | Spec? | Lower lim | Upper lim | Lower lim Upper lim Book-Data Analyses | Analyst   | Verifier |
|------------|------------------------|---------|-----------|-------|-----------|-----------|--|-----------|----------|
| 08360801#0 | SAMPLE#1               | 96.29   | <br> <br> |       | 80.000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            |                        | 95.75   | *         |       | 80.000    |           | 15545/18-MAY 2002                      | CAPPUCCIO | MARIANI  |
|            | SAMPLE#3               | 96.82   | %         |       | 80,000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            | SAMPLE#4               | 100.05  | %         |       | 80.000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            | SAMPLE#5               | 99.51   | *         |       | 80.000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            | SAMPLE#6               | 97.90   | %         |       | 80.000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            | DISSOLUTION            | 97.72   | %         |       | 80.000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            | STANDARD DEVIATION     | 1.755   | \$ld-1    |       |           |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIAN   |
|            | Ö.V.                   | 1.8     | %         |       |           |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |
|            | MIN. DISSOLUTION VALUE | 95,75   | 96        |       | 80.000    |           | 15545/18- MAY 2002                     | CAPRUCCIO | MARIANI  |
|            | MAX, DISSOLUTION VALUE | 100.05  | %         |       | 80.000    |           | 15545/18- MAY 2002                     | CAPPUCCIO | MARIANI  |



Integrated Analytical Bulletin

Management Information Laboratory System

#### Pharmaceutical Development

Submission Id: 116307

Sample ID (Request No.): 20021136 User sample ID (lot): 183GÓ3

Sample Date: 18/04/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE

25MG

Batch Manufacture Date: 17/09/2001 Stability Study: 183G03

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 40°C/75% U.R. Time Label: 6 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

condition: APPROVED

COMPLETE Status:

Stabilly Study Start Date: 18/10/2001 Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano

Samples Notes:

083GC002#00, component: CAPSULE APPEARANCE Observations on the Method

Opaque brick red colored, format 3, hard gelatin capsule containing red-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Dissolution at 15 minutes: 96.74% (C.V. = 1,77%)

Dissolution at 45 minutes; 97.72% (C.V. = 1.86%)

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

Total correlated substances: 0.21%

Area (%) RRT

0.08

0.07 99.79 0.66 0.91 1.00

(SU11248)

17-may-2002 Approved by: MARIANI Approval date:

Printed



Integrated Analytical Bulletin

Analytical Bulletin

Laboratory Information Management System

#### Pharmaceutical Development

Submission Id: 115471 Sample ID (Request No.),20020165 User sample ID (Iot) 183G03 Sample Date: 18/01/2002

Sample Type: SU010338 (PNU290940AD) CAPSULE 25MG

ability Study Start Date: 18/10/2001

Request Type: RELEASE Stability Study: 183G03 Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25AC/60% U.R.

Batch Manufacture Date: 17/09/2001

Time Labet: 3 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition:
APPROVED
Status:
COMPLETE

|                              | cia Nerviano   |
|------------------------------|--|
|                              | tocol Number: 2001017 - Manufacturer: PD/Pharmacia Ner |
|                              | irer: PD/F   |
| 002010                       | Manufactu  |
| 5                            | ₹  |
| _                            | Ξ  |
|                              | Ħ.   |
| ē                            | ន្ត  |
| ב                            | :  |
|                              | 효  |
| 2                            | ₫  |
| g                            | Z.   |
| 5                            | ĕ  |
| Stability Study Start Date : | Stability Protoco                                      |
| 景                            | <u>ъ</u>   |
| 2                            | ≝  |
| "                            | 쳝  |
|                              | u)   |

| Method       | Annual of the Party of the Part | The second secon |      |       |                     |                    | The second secon |          |
|--------------|--|--|------|-------|---------------------|--------------------|--|----------|
|              | Controls   | Results  | M.U. | Spec? | Lower lim Upper lim | Book-Data Analyses | Analyst  | Verifier |
| 083GCO02#0 . | 083GCO02#0 . CAPSULE APPEARANCE  | 0=UNCHANGED  | ¥    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
| 0            | CAPSULE COLOR  | 0=UNCHANGED  | ¥    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCAR   |
|              | CONTENT APPEARANCE   | 0=UNCHANGED  | ¥    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
|              | CONTENT COLOR  | 0=UNCHANGED  | Ħ    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
|              | SMELL  | 0=UNCHANGED  | ¥    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
| 083GTI01#01  | (HPLC TITER) SAMPLE#1  | 98.84  | ×    |       | 90.000 - 110.000    | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
| ما<br>م      | (HPLC TITER) SAMPLE#2.   | 96.31  | %    |       | 90,000 - 110,000    | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
|              | (HPLC TITER) SAMPLE#3  | 96.41  | *    |       | 90.000 - 110.000    | · 143/44- JAN 2002 | CAPPUCCIO  | MACCARI  |
|              | AVERAGE TITER (HPLC)   | 97.19  | %    |       | 90.000 - 110.000    | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
| 083GSC01#0   | TOTAL CORRELATED SUBSTANCES 0.21   | 0.21   | %    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
| 0            | IMPURITIES NOT LESS THAN 0.05%   | SEE TEXT   | *    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
| 083GKF01#00  | (HUMIDITY) SAMPLE #1   | 1.9  | 96   |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
|              | (HUMIDITY) SAMPLE #2   | 1.82   | %    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
|              | (HUMIDITY) SAMPLE#3  | 1.9  | %    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |
|              | AVERAGE HUMIDITY   | 1.87   | %    |       |                     | 143/44- JAN 2002   | CAPPUCCIO  | MACCARI  |

Printed



## Integrated Analytical Bulletin

Management System Laboratory Information

#### Pharmaceutical Development

Sample ID (Request No.): 20020165 Submission Id: 115471

Sample Date: 18/01/2002 User sample ID (lot): 183G03

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Time Labet: 3 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/C/60% U.R. Request Type: RELEASE Stability Study: 183G03

Batch Manufacture Date: 17/09/2001

condition: APPROVED

COMPLETE Status:

| Method     | Controls               | Results | M.U.       | Spec7 | Lower lim | Upper lim | Lower lim Upper lim Book-Data Analyses | Analyst   | Verifier |
|------------|------------------------|---------|------------|-------|-----------|-----------|--|-----------|----------|
| 083GDS01#0 | SAMPLE#1               | 101.14  | %          |       | 80,000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
| -          | SAMPLE#2               | 106.48  | %          |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | SAMPLE#3               | 109.97  | %          |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | SAMPLE#4               | 106.90  | <b>3</b> % |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | SAMPLE#5               | 105.33  | *          |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | SAMPLE#6               | 104.08  | %          |       | 80,000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | DISSOLUTION            | 105.65  | %          |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | STANDARD DEVIATION     | 2.96    | std-1      | •     |           |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | c.v.                   | 89      | %          |       |           |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | MIN. DISSOLUTION VALUE | 101.14  | *          |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |
|            | MAX. DISSOLUTION VALUE | 109,97  | *          |       | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO | MACCARI  |

Printed



### Integrated Analytical Bulletin

Management Information Laboratory System

#### Pharmaceutical Development

Sample ID (Request No.): 20020165 Submission Id: 115471

User sample ID (lot): 183G03

Sample Date: 18/01/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Stability Study. Start Date: 18/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano

Batch Manufacture Date: 17/09/2001 Request Type: RELEASE

Pack Type: 30 ml PLASTIC BOTTLE Stability Study: 183G03

Time Label: 3 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY Starage Condition: 25/C/60% U.R.

Condition: APPROVED

COMPLETE Status:

Samples Notes:

083GCO02#00, component: CAPSULE APPEARANCE Observations on the Method

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

083GDS01#01, component: DISSOLUTION Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

Total correlated substances: 0.21%

Area (%) HHT

(SU 11248) 99.79 0.09 0.12 1.00 1.97 2.06

04-feb-2002 Approved by: MACCARI Approval date:

by MARIAN!



Management Information Laboratory

System

### Integrated Analytical Bulletin

#### Pharmaceutical Development

Sample ID (Request No.): 20020167 User sample ID (lot): 183G03 Submission Id: 115473

Semple Type: SU010398 (PNU290940AD)CAPSULE 25MG Sample Date: 18/01/2002

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano Samples Notes: Stability Study Start Date: 18/10/2001

Stability Study: 183G03
Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 40°C/75% U.R.

Batch Manufacture Date: 17/09/2001

Request Type: RELEASE

Condition: APPROVED

Status: COMPLETE

Time Label: 3 MONTHS
Preparation purpose: CLINICAL SUPPLY + STABILITY

|  | Verifier   | MACCARI                              |
|--|--|--------------------------------------|
|  | Analyst  | CAPPUCCIO                            |
|  | Results M.U. Spec? Lower lim Upper lim Book-Data Analyses Analyst Verifier | 143/44- JAN 2002<br>143/44- JAN 2002 |
| COCOCCO  | Lower Iim Upper Iim  |                                      |
|  | Spec?  |                                      |
| Statement of the last  | M.U.   | * *                                  |
| Company of the last of the las | Results  | 0=UNCHANGED<br>0≕UNCHANGED           |
|  |  | CAPSULE APPEARANCE<br>CAPSULE COLOR  |
| -  | Method Controls  | 083GCO02#0<br>0                      |

| Rethod         | Controls                               | Results     | M.U.       | Spec? | Lower lim | Upper lim        | Spec? Lower lim Upper lim Book-Data Analyses | Analyst   | Verifier |
|----------------|--|-------------|------------|-------|-----------|------------------|--|-----------|----------|
| 083GCO02#0     | 083GCO02#0 CAPSULE APPEARANCE          | 0=UNCHANGED | ¥          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
| 0              | CAPSULE COLOR                          | D≂UNCHANGED | Ħ          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
|                | CONTENT APPEARANCE                     | 0=UNCHANGED | Ħ          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
|                | CONTENT COLOR                          | 0=UNCHANGED | Ħ          |       |           |                  | 143/44 JAN 2002                              | CAPPUCCIO | MACCARI  |
|                | SMELL                                  | 0=UNCHANGED | 뀲          |       |           |                  | 143/44-JAN 2002                              | CAPPUCCIO | MACCARI  |
| 083GTI01#01    | 083GTI01#01 (HPLC TITER) SAMPLE#1      | 99.21       | *          |       | 90.000    | 90,000 - 110,000 | 143/44-JAN 2002                              | CAPPUCCIO | MACCARI  |
| ω <sub>I</sub> | (HPLC TITER) SAMPLE#2                  | 96.18       | %          |       | 90.000    | 90.000 - 110.000 | 143/44-JAN 2002                              | CAPPUCCIO | MACCARI  |
|                | (HPLC TITER) SAMPLE#3                  | 95.82       | *          |       | 90.000    | 90.000 - 110.000 | 143/44-JAN 2002                              | CAPPUCCIO | MACCARI  |
|                | AVERAGE TITER (HPLC)                   | 20.78       | *          |       | 000'06    | 90,000 - 110,000 | 143/44~JAN 2002                              | CAPPUCCIO | MACCARI  |
| 083GSC01#0     | 083GSC01#0 TOTAL CORRELATED SUBSTANCES | 0.41        | <b>3</b> € |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
| <b>.</b>       | IMPURITIES NOT LESS THAN 0.05%         | SEE TEXT    | %          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
| 083GKF01#00    | 083GKF01#00 (HUMIDITY) SAMPLE #1       | 1.8         | %          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
|                | (HUMIDITY) SAMPLE #2                   | 1.7         | %          |       |           | -                | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
|                | (HUMIDITY) SAMPLE #3                   | 1.9         | %          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |
|                | AVERAGE HUMIDITY                       | 8.          | %          |       |           |                  | 143/44- JAN 2002                             | CAPPUCCIO | MACCARI  |

svíbollaintegrafed.rdf

20-MAR-2002 05:47 PM by

Printed

MARIANI

Page



Integrated Analytical Bulletin

Management System Laboratory Information

#### Pharmaceutical Development

Sample ID (Request No.): 20020167 Submission Id: 115473

User sample ID (lot): 183G03

Sample Date: 18/01/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Stability Study Start Date: 18/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano Samples Notes:

Pack Type: 30 ml PLASTIC BOTTLE Batch Manufacture Date: 17/09/2001 Request Type: RELEASE Stability Study: 183G03

Preparation purpose: CLINICAL SUPPLY + STABILITY Storage Condition: 40vC/75% U.R. Time Label: 3 MONTHS

Condition: APPROVED

Status: COMPLETE

| Method     | Controls               | Results | M.U.  | Spec | Lower lim | Upper lim | Lower lim Upper lim Book-Data Analyses | Analyst     | Verifier |
|------------|------------------------|---------|-------|------|-----------|-----------|--|-------------|----------|
|            |                        |         |       |      |           |           |  |             |          |
| 083GDS01#0 | SAMPLE#1               | 101.47  | %     |      | 80,000    |           | 143/44-JAN 2002                        | CAPPUCCIO   | MACCARI  |
|            | SAMPLE#2               | 102.93  | *     |      | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | SAMPLE#3               | 104.56  | *     |      | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO · | MACCARI  |
|            | SAMPLE#4               | 106.96  | 38    |      | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
| •          | SAMPLE#5               | 101.34  | *     |      | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | SAMPLE#6               | 98.51   | %     |      | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | DISSOLUTION            | 102.63  | %     |      | 80.000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | STANDARD DEVIATION     | 2.917   | std-1 |      |           |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | C.V.                   | 2.84    | *     |      |           |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | MIN. DISSOLUTION VALUE | 98.51   | *     |      | 80,000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |
|            | MAX. DISSOLUTION VALUE | 106.96  | %     |      | 80,000    |           | 143/44- JAN 2002                       | CAPPUCCIO   | MACCARI  |

Printed



## Integrated Analytical Bulletin

Management System Information Laboratory

#### Pharmaceutical Development

Submission Id: 115473

Sample ID (Request No.): 20020167

User sample ID (lot): 183G03

Sample Date: 18/01/2002

Sample Type: SU010398 (PNU290940AD) CAPSULE 25MG

Stability Study Start Date: 18/10/2001

Batch Manufacture Date: 17/09/2001 Request Type: RELEASE

Pack Type: 30 ml PLASTIC BOTTLE Stability Study: 183G03

Storage Candillon: 40°C/75% U.R. Time Label: 3 MONTHS

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Stability Protocol Number: 2001017 - Manufacturer: PD/Pharmacia Nerviano Samples Notes:

083GCO02#00, component: CAPSULE APPEARANCE Observations on the Method

Opaque brick red colored, format 3, hard gelatin capsule containing yellow-orange powder

Observations on the Method 083GDS01#01, component: DISSOLUTION

Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.41%

Area (%) FE 0.05 99.60 (SU 11248) 0.07 0.29 0.94 1.00 1.97 2.06

Approved by: MACCARI

Approval date: 04-feb-2002

by MARIANI



Pharmacia & Upjohn
Pharmaceutical Development

Integrated Analytical Bulletin

Laboratory Information Management System

Submission id: 115029

Sample ID (Request No.): 20013360

Sample Date: 15/11/2001 User sample ID (lot): 183G03

Semple Type: SLOGGES (PALZEDADAD) CAPSULE ZEMS
Assurt Date: 1810/2011

Satch Manefecture Date; 17/09/2001 Stability Study: 183500

Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25°C/80% U.R.
Time Label: 1 MONTH

APPROVED STATES COMPLETE

Preparation purpose: CLINICAL SUPPLY + STABILITY

| Method Controls Results M.U. Spec? Lower lim Upper lim Book-Data Analyses Analyst | Samples Notes: | lotes.   |         |      |       |           |           |                    |         |          |
|---|----------------|----------|---------|------|-------|-----------|-----------|--------------------|---------|----------|
|   | Method         | Controls | Results | M.U. | Spec? | Lower lim | Upper lim | Book-Data Analyses | Analyst | Verifier |

| Method         | Controls                            | Results       | M.U. | Spec? | Lower lim        | Upper lim       | Book-Data Analyses | Analyst    | lyst Verifier |
|----------------|-------------------------------------|---------------|------|-------|------------------|-----------------|--------------------|------------|---------------|
| disaccocato    | GIBSCOCKIO CAPSULE APPEARANCE       | CONFORMS      | ¥    |       |                  | HONOR           | 143/20 NOV 2001    | CAPPUCCIO  | MACCAR        |
| <b>-</b>       | CAPSULE COLOR                       | CONFORMS      | Ħ    |       |                  | 143/2017        | 14\$V20 NOV 2001   | CAPPUCCIO  | MACCAR        |
|                | CONTENT APPEARANCE                  | CONFORMS      | ¥    |       |                  | 143/20 N        | 143/20 NOV 2001    | CAPPUCCIO  | MACCAR        |
|                | CONTENT COLOR                       | CONFORMS      | 뀰    |       |                  | 143/20 N        | 145/20 NOV 2001    | CAPPUCCIO  | NUCCAR        |
|                | SMELL                               | CONFORMS      | Ħ    |       |                  | 143/20 N        | 14S/ZO NOV ZOOI    | CAPPUCCIO  | MACCAR        |
| OBSCTICANOL    | OGSGTICATOL (HPLC TITER) SAMPLE#1 8 | 98.783        | 3E   | Š     | 90,000 - 110,000 |                 | 143/20 NISV 2001   | CAPPUCCIO  | MOCAR         |
| a <sub>l</sub> | (HPLC TITER) SAMPLE#2 9             | 96.216        | ×    | 76    | 90,000 - 110,000 |                 | HAZEO HOV 2004     | CAFFUCCIO  | MACCAR        |
|                | (HPLC TITER) SAMPLE#3               | 95.918        | 32   | g     | 92.000 - 119.000 |                 | H320 HOV 2001      | CAPPLICCIO | MACCAR        |
|                | AVERAGE TITER (HPLC) 8              | 86.3          | *    | 8     | 90,000 - 110,000 |                 | 148/20 MOV 2001    | CAPPUDCIO  | MACCARE       |
| 0836900140     | TOTAL CORRELATED SUBSTANCES         | 0.306         | *    |       |                  | 143/201         | 14820 NOV 2001     | CAPPLUCCIO | MACCAR        |
| •              | S THAN                              | 0.05% SEETEXT | *    |       |                  | 143/20 K        | 143/20 NOV 2001    | CAPPLICOLO | IMCCAR        |
| 083CKT01#00    | ASSKRUTMOD (HUMIDITY) SAMPLE#1      |               | ×    |       |                  | 143/20 N        | 443/20 NOV 2014    | CAPPUCCIO  | MACCAR        |
|                | (HUMIDITY) SAMPLE #2                | 1.6           | ×    | -     |                  | 143/20 N        | 143/20 NOV 2001    | CAPPUDGIO  | IMOCAR        |
|                | (HUMIDITY) SAMPLE#3                 | יז            | r.   |       |                  | 143/20 NOV 2001 | IOV 2001           | CAPPUCCIO  | MACCAR        |
|                | AVERAGE HUMIDITY 1                  | ב             | *    |       |                  | 143/20 N        | 143/20 NOV 2001    | CAPPUCCEO  | MACCAR        |

Page



Latoretory Information Management System

Constition: APPROVED

SOMPLETE

Sample ID (Request No.); 20013300 User sample ID (lot): 163G13

Sample Date: 1871/2001

Sample Type: SU010306 (PNU2010410A)) CAPSULE 25MG

Preparation purpose: CLINICAL SUPPLY + STABILITY

The Label: 1 MONTH

Protryper: 30 ml PLASTIC BOTTLE Borage Cardifon: 25/CR0% U.R.

Betch Manyfacture Dafe: 17/09/2001

Stability Study, 183603

Stabilly Study Start Date: 18710/2001

Statuly Protocol Number: 2001017 - Manufacturer: POWframencia Nerviewo

| -            |                            |         |      |       |        |  |            |          |
|--------------|----------------------------|---------|------|-------|--------|--|------------|----------|
| Method       | Controls                   | Results | M.U. | Spec? | 1      | Lower lim Upper lim Book-Data Analyses | Analyst    | Verifier |
| MESCENSO (#0 | DESCISO (40 (SAMPLE #1     | 258     | ¥    |       | 80.000 | 143/20 NOV 2201                        | CAPPUCCIO  | MACCAR   |
|              | (SAMPLE #2                 | 84.8    | ×    |       | 9000   | 143/20 NOV 2001                        | CAPPUCCID  | MACCAR   |
|              | (SAMPLE #3                 | 424     | *    |       | 2000   | 143/20 NOV 2001                        | CAPPUCCIO  | MACCAR   |
|              | SAMPLE #4                  | 68.0    | *    |       | 80,000 | \$43/20 NOV 2804                       | CAPPUCCIO  | MACCARI  |
|              | SAMPLE #5                  | 430     | *    |       | 89,000 | 14320 NOV 2001                         | CAPPUCCIO  | MACCAR   |
|              | SAMPLE #6                  | 54.7    | ×    |       | 90,000 | 14 a 2 b N D V 2001                    | CAPPUCCIO  | MACCAR   |
|              | PISSOLUTION                | 90.36   | 試    |       | 88/050 | 44.820 NOV 2001                        | CAPPUOCIQ  | HACCAR   |
|              | STANDARD DEVIATION         | 525     | ¥    |       |        | 143/20 NOV 2001                        | CAPPLICGIO | MACCAR   |
|              | Ć.V.                       | 5.81    | *    |       |        | 443726 NOV 2001                        | CAPPUCCIO  | MACCAPA  |
|              | IMINIMUM DISSOLUTION VALUE | 61.8    | ×    |       | 60.000 | 143/20 NOV 2001                        | CAPPLICAO  | MOCHE    |
|              | IMAXIMUM DISSOLUTION VALUE | 96.24   | ×    |       | 90,000 | 143/20 NOV 2001                        | CAPPUCCIO  | MADCAR   |

Page



Pharmacia & Upjohn

Integrated Analytical Bulletin

Laborakoy Information Management System

Pharmaceutical Development

Sample ID (Request No.): 20013300 Submission Mr. 113029

User sample ID (lot): #3GD3

Sample Date: 18/11/2001

Sample Type: SULHOSBS (PNU2SOCKOAD) CAPSULE 28MG

Balich Manufacture Date: 17/09/2001 Stability Study: 185603

Time Label: (MCNTH)
Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Storage Condition: 25/0/80% U.R.

CONTINUES:
APPROVED STATE COMPLETE

Stability Study Start Date: 18/10/2017

Stability Psobool Number (2001937 - Manifecturer: POP harmon Nerviews Samples Notes:

Observations on the Method 083GCO02#00, component: CAPSULE APPEARANCE Brick red colored capsule containing yellow-orange powder

Observations on the Method

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.306%

(SV) 11(246) Acoa X 0.090 09.90 0.076 0.140 돭 

MACCARI 30-new-2001 Approved by:

Approval date:



### Pharmacia & Upjohn

#### Integrated Analytical Bulletin

Laboratory Information Management System

Pharmaceutical Development

Sample ID (Request No.): 20013302 User sample ID (lot): 183G03 Sabralesion id: 115031

Sample Type: SU010898 (PNU200960/D)CAPSULE 25MG Sample Date: 18/11/2001

Batch Manufactore Date: 17/09/2001 Simbliffy Mandy: 182903

Pack Types 30 ml PLASTIC BOTTLE
##Krage Conditions 40/C/75% UR.
Time Label: 1 MONTH
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Statisty Protocial Number : 2001017 - Danufacturer : PDFFrematics Norview Samples Notes:

| Method        | Controls                                   | Results  | M.U. | Spec? | Results M.U. Spec? Lower lim Upper lim Book-Data Analyses | Upper lim | Book-Data Analyses | Analyst    | yst Verifier |
|---------------|--|----------|------|-------|---|-----------|--------------------|------------|--------------|
| DESCODE       | OUSCOUDE CAPSULE APPEARANCE                | CONFORMS | B    |       |   | 14320     | 4320 NOV 2001      | CAPPUCCID  | MACCAR       |
| •             | CAPSULE COLOR                              | CONFORMS | B    |       |   | 143/20    | HAZD NOV 2001      | CAPPUCCEO  | MACCARI      |
|               | CONTENT APPEARANCE                         | CONFORMS | R    |       |   | 143/20    | 19320 NOA 2001     | CAPFUECIO  | MACCARI      |
|               | CONTENT COLOR                              | CONFORMS | 3    |       |   | 143720    | 143/20 INOV 2001   | CAPPLICCIO | SAACCAR      |
|               | SMELL                                      | CONFORMS | 3    |       |   | 143/20    | 143/20 NOV 2001    | CAPPUCCIO  | MOCAR        |
| ORBESTRO LATO | OGSGTTO1#01 (HPLC TITER) SAMPLE#1          | 87.4B    | ×    | ă     | 20,000 - 110,000  |           | 143020 NOV 2001    | CAPPUCCIO  | MACCAR       |
| <b>1</b> 2    | (HPLC TITER) SAMPLE#2                      | 80°03    | ×    | 86    | 90,000 - 110,000  |           | 14325 NOV 2001     | CAPPUCCIO  | MACCAR       |
|               | (HPLC TITER) SAMPLE#3                      | 96.23    | *    | 85    | 90,000 - 110,000  |           | 14325 NOY 2011     | CAPPUCCIO  | MACCAR       |
|               | , AVERAGE TITER (HPLC)                     | 97,25    | ×    | 35    | 90,000 - 110,000  |           | 143/20 NOY 2017    | CAPPUCCIO  | MACCAR       |
| 083GSCD14     | OBJESSION TOTAL CORRELATED SUBSTANCES 6.15 | 0.15     | *    |       |   | 143/20    | Haza Non Zedi      | CAPPUDGIO  | MACCAR       |
|               | IMPURITIES NOT LESS THAN 0.05%             | SEE TEXT | *    |       |   | 143/20    | 143/20 NOV 20th    | CAPPUCCIO  | MACCAFE      |
| 063GKT-01#    | OCCURTOHADO(HUMIDITY) SAMPLE #1            | 1.8      | ×    |       |   | 143/20    | 14320 NOV 2451     | CAPPUCCIO  | MMOCAR       |
|               | (HUMIDITY) SAMPLE #2                       | 4.9      | ×    |       |   | 143/20    | 143/23 NOV 2001    | CAPPUCCIO  | MADCAR       |
|               | (HUMIDITY) SAMPLE #3                       | 1.8      | *    |       |   | 143720    | 143250 NOV 2001    | CAPPUCCIO  | MACCAR       |
|               | AVERAGE HUMIDITY                           | 1.83     | ×    |       | ,   | 143/20    | 143/20 NOV 2001    | CAPPUCCID  | MACCAR       |



Laboratory Information Management System

Submission 1d: 115031

Sample ID (Request No.); 20013302

User sample ID (lot): **\$33003** 

Semple Dale: 187112001

Semple Type: SUC10308 (PNU28004GAD) CAPSULE 25MG

Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Types so mi PLASTIC BOTTLE Storage Conditions APC/75% U.R.
Time Label: 1 MONTH

Condition: APPROVED

Baich Wannincture Dale: 17/09/2001 Stability Shudy: R3G03 SMAN: COMPLETE

Saabilly Study Start Clate: 16/10/2001

Stability Protocol Number: 2001017 - Manufacturer: POPPramacia Neroleno

| - 6 |     |
|-----|-----|
| h   | ::: |
| 21  | *   |
| 3   | 22  |
| •   | 0   |
| M   | ~   |
| 13  |     |
| 4   | tr) |
|     | qu. |
|     | 75  |
| 3   | =   |
| -   | ┶   |
| 3   | ₩.  |
| 6   | Ö   |
| п   | ٠,  |
|     |     |

| lethod   | Controls Results          | Results | M.U.  | Spec? | pec? Lower lim Upper li | Upper lim | Lower lim Upper lim Book-Data Analyses | Analyst    | Verifier | 1 |
|----------|---------------------------|---------|-------|-------|-------------------------|-----------|--|------------|----------|---|
| SGDS01#0 | ENGENERALE #1             | Į.      | *     |       | 80,000                  |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCAR   |   |
|          | SAMPLE#2                  | 8       | ×     |       | 80,000                  |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCAR   |   |
|          | SAMPLE#3                  | 862     | ×     |       | 80,000                  |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCAR   |   |
|          | SAMPLE#4                  | 67.5    | ×     |       | 60,000                  |           | 143/20 NCV 2001                        | CAPPLICCID | MACCARI  |   |
|          | SAMPLE #5                 | 87.3    | ×     |       | 80,000                  |           | 143/20 MCV 2001                        | CAPPUCCIO  | MACCAR   |   |
|          | SAMPLE #6                 | 97.3    | *     |       | 00000                   |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCARI  |   |
|          | DISSOLUTION               | 853     | ¥     |       | 90,090                  |           | 145/20 NOV 2001                        | CAPPLICCIO | MACCAR   |   |
|          | STANDARD DEVIATION        | 4.182   | 1-046 |       |                         |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCAR   |   |
|          | C.V.                      | 4.38    | ×     |       |                         |           | 143/20 MOV 2001                        | CAPPUCCIO  | MACCAPI  |   |
|          | MINIMUM DISSOLUTION VALUE | 87.5    | ×     |       | 60,000                  |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCAR   |   |
|          | MAXIMUM DISSOLUTION VALUE | 98.33   | ¥     |       | 60,000                  |           | 143/20 NOV 2001                        | CAPPUCCIO  | MACCARR  |   |



Pharmacia & Upjohn

Integrated Analytical Bulletin

Laboratory information Management System

Sample ID (Request No.); 20013302

Submission Id: 115031

User sample ID (lot): **163Gd3** 

Serupte Type: SU010308 (PNIX2809404D)CAPSULE 2584G Sample Date: 18/11/2001

Besitzi Mensufathan Date: 1700/2007

Storage Condition: 40'G75% U.R.
Thes Labor 1 MCNTH
Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: 30 ml PLASTIC BOTTLE Shibility Shidy, 85503

APPROVED SCOMPLETE

Stability Start Start Date: 18/10/2014

Statility Protocol Number: 2001017 - Manufacture: POR hermacia Marvino

Samples Notes:

Observations on the Method 083GC002#00, component: CAPSULE APPEARANCE

Brick red colored capsule containing yellow-orange powder

083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05% Observations on the Method

Total correlated substances: 0.145%

Area X Ē

99,66 (SU-11248) 0.49 1.00 98

Approved by: WACCARI Approval date: 30-nov-2001

Page



Laboratory Information Menegement System

Pharmaceutical Development

Sample Date: D151/2001 Sample ID (Request No.): 20013158 User sample ID (lot): ISSGGS Submission Id. 114005

Sample Type: SUD10388 (PMU290940AD) CAPSULE 25M3

Storage Contilion: TME ZERO Time Label: TIME ZERO Bertin Manufactors Date: 17/09/2001 State May Sturby: 153503 Pack Type: BLLK

Preparation purpose: CLINICAL SUPPLY + STABILITY

CONCEIN:
APPROVED

Status: COMPLETE

Stability Protocol Namber: 2001017 - Manylacture: POPPiermane Nervieno
Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder Shibility Study Start Date: 19/10/2001

| Method          | Controls                                | Results   | M.U. | Spec7 | Lower lim        | Upper lim | Lower lim Upper lim Book-Data Analyses | Analyst    | Verifier |
|-----------------|---|-----------|------|-------|------------------|-----------|--|------------|----------|
| 6839COO1#0<br>1 | 0330COOMA APPEARANCE                    | COMPLIANT | ¥    |       |                  |           | 143KN OCT 2091                         | CAPPUCCIO  | NACCAR   |
| 483CBZ41801     | GBSGZG11001 HPLC IDENTIFICATION         | POSITIVE  | Ħ    |       |                  |           | 143KH OCT 2001                         | CAPPUCCIO  | NACCAR   |
| DB3GTIG UNOT    | 083GT/01001 (HPLC TITER) SAMPLE#1       | 97.3t     | ×    |       | 95,000 - 105,000 | 105.000   | 142/01 OCT 2001                        | CAPPUCCIÓ  | MACCAR   |
| <b>≃</b> ,      | (HPLC TITER) SAMPLE#2                   | 99.10     | ×    |       | 95,000 - 105,000 | 105,000   | 143/c1 OCT 2001                        | CAPPLUCCID | MACCARD  |
| •               | (HPLC TITER) SAMPLE#3                   | 78.87     | *    |       | 95,000 - 165,000 | 185.000   | 143/th OCT 200/                        | CAPPLUCIO  | MACCAR   |
|                 | AVERAGE TITER (HPLC)                    | 60188     | ¥    |       | 95,000 - 105,000 | 000301    | 143/01 OCT 2001                        | CAPPUCCIO  | MACCAR   |
| DESCOUND        | DROGSCOMMO TOTAL CORRELATED SUBSTANCES  | 0.zz3     | ×    |       |                  |           | 143/01 OCT 2004                        | CAPPUCCIO  | MACCAR   |
| <b>5</b>        | IMPURITIES NOT LESS THAN 0.05% SEE TEXT | SEE TEXT  | ×    |       |                  |           | 143/bf OCT 2001                        | CAPPUCCIO  | MACCAR   |
| DESCRIPTION     | DESCREPATION (HUMIDITY) SAMPLE #1       | 1.85      | ×    |       |                  |           | 143/51 OCT 2001                        | CAPPLICCED | MACCAR   |
|                 | (HUMIDITY) SAMPLE #2                    | 1.78      | *    |       |                  |           | 143/0H OCT 2001                        | CAPPUCCIO  | NACCAR   |
|                 | (HUMIDITY) SAMPLE #3                    | 1.90      | *    |       |                  |           | 142401 OCT 2001                        | CAPPUDGIO  | MACCAR   |
|                 | HUMIDITY) SAMPLE                        | 1.9       | Ж    |       |                  |           | 143/01 OCT 2001                        | CAPPLICATO | MACCAR   |

Page 1 / 6



Laboratory Information Management System

Submission Id: 114905

Sample ID (Request No.); 20013158 User sample ID (lot): 183/G03 Semple Date: 01/11/2001

Stability Shady Start Date: 18/10/2001

Surrpive Types: SUO10080 (PNU280840AD) CAPSULE 2514G

Batch Manufacture Date : 17/09/2001 Stabilly Stady: 183003

Storage Condition: TIME ZERO Thes Lebel: TIME ZERO Puck Type: BULK

Condition: APPROVED Statue: COMPLETE

Preparation purpose: CLINICAL SUPPLY + STABILITY

Stability Protocol Number: 2001617 - Manefacturer: POPPtannada Nevitano
Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder

| Method Controls |                           | Results | M.U. Spec?     | Lower lim | Upper IIm Book-Data Analyses | es Analyst | Verifier |
|-----------------|---------------------------|---------|----------------|-----------|------------------------------|------------|----------|
| 063GDS01#0      | DESIGNOTING SAMPLE#1      | 85.23   | æ              | 90.000    | MANY OCT ZON                 | CAPPLICATO | MACAN    |
| 7               | SAMPLE #2                 | 9279    | ×              | 80,000    | 14301 OCT 2001               | CAPPLICCIO | MACCAR   |
|                 | SAMPLE #3                 | 38.54   | *              | 90,000    | 143/01 OCT 2001              | CAPPUCCIO  | MACCAR   |
|                 | SAMPLE #4                 | 93.86   | ×              | 90,000    | HAIDS OCT ZADI               | CAPPUCCIO  | MACCARI  |
|                 | SAMPLE #5                 | 84.52 · | *              | 80,000    | 143/D1 OCT 2001              | CAPPUCCIO  | MACCARI  |
|                 | SAMPLE #6                 | 84.74   | 3 <sup>8</sup> | 90,000    | 14301 OCT 2001               | CAPPUCCIO  | MACCHE   |
|                 | SAMPLE #7                 | 88.32   | ×              | 80,000    | M301 OCT 2001                | CAPPLOCIG  | MACCAR   |
|                 | SAMPLE#8                  | 95.91   | ¥              | 60,000    | 143TH OCT 2001               | CAPPLICATO | MACCAR   |
|                 | SAMPLE #9                 | \$90,6% | *              | 00000     | 14301 OCT 2001               | CAPPUCCIO  | · MACCAR |
|                 | SAMPLE #10                | 106.5   | *              | 00000     | Habt oct 2001                | CAPPLICCIO | MACCAR   |
|                 | SAMPLE #11                | 96.31   | ×              | 9000      | HANN OCT 2001                | CAPRUCCIO  | MADCAR   |
|                 | SAMPLE #12                | 88.12   | *              | 80,000    | 14301 OCT 2001               | CAPPLICCIO | MADCAR   |
|                 | DISSOLUTION               | 29.28   | ઝ              | 00000     | 44304 OCT 2001               | CAPPLICCIO | MADCAR   |
|                 | STANDARD DEVIATION        | 7,031   | THE            |           | 443101 OCT 2001              | CAPPUCCIO  | MADCAR   |
|                 | C.V.                      | 7.59    | ¥              |           | 143th OCT 2001               | CAPPUCCIO  | MACCAR   |
|                 | MINIMUM DISSOLUTION VALUE | 81,62   | ×              | 900'09    | 14301 OCT 2001               | CAPPUCCIO  | MACCAR   |
|                 | MAXIMUM DISSOLUTION VALUE | 105.5   | ×              | 80,000    | M3101 OCT 2001               | CAPPLICCIO | MACCAR   |



Laboratory Information Management System

Schmisskas td: 114908

Sample ID (Request No.): 20013458 User sample ID (lot); #35563

Sample Date: 01/11/2001

Semple Type: SUGGOSS (PNUZSUSGAD) CAPSULE 25545

Batch Manufacture Date: 17/09/2001 Stahilly Study: 163603

Shorage Continuous, TMIEZERO
Time Label: TileEZERO
Preparation purpose: CLINICAL SUPPLY + STABILITY Pack Type: BULK

CONTENT STATE APPROVED Status: COMPLETE

> Sanding Protocol Number : 2001017 - Washindurer: POP hamada Nardano Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder Statement Starty Start Date : 18/10/2001

| Method      | Method Controls Results M.U. Spec? Lower lim Upper lim Book-Data Analyses Analyst Verifier | Results   | M.U. | Spec? | Lower lim | Upper lim | Book-Data Analyses | Analyst    | Verifier |
|-------------|--|-----------|------|-------|-----------|-----------|--------------------|------------|----------|
| OBSCHOOLING | UBECINCOLING TOTAL AEROBIC BACTERIA  | IN LIMITS | ¥    |       |           |           | 14301 OCT 2301     | CAPPLICCIO | NACCAR   |
| -           | ESCHERICHIA COLI   | ABSENT    | 3    |       |           |           | 142/01 OCT 2001    | CAPPUCCIO  | MACCAR   |
|             | SALMONELLA   | ABSENT    | Ħ    |       |           |           | 143/01 OCT 2001    | CAPPUCCIO  | NACCAR   |
|             | FUNGI  | INLIMITS  | Ħ    |       |           |           | 14301 OCT 2001     | CAPPUCCIO  | HADCAR   |
|             | STAPHYLOCOCCUS AUREUS  | ABSENT    | Ħ    |       |           |           | 143/01 OCT 2001    | CAPPUCCIO  | HADCAR   |
|             | ENTEROBACTERIA   | IN LIMITS | ¥    |       |           |           | 143/bt OCT 2001    | CAPPUCCIO  | MAGGARI  |

3 6

Page



Laboratory Information Menagement System

Sample ID (Request No.); 20013138 User sample ID (lot), **195563** Sabmission M: 114806

Sangile Type: SUDJOSGE (PMI230340AD) CAPSULE 2346 Semple Dade: 01/11/2001

Batch Mandisches Date: 17/09/2001 Statute Study: 183503

Prack Type: BLUK
Shorage Conditions The EXERC
Time Label: THE ZERO
Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

Status: COMPLETE

Shaldling Stody Start Date: 19/10/2001

Stability Protocol Number: 2001017 - Manufacturer: PDR transpora Nervieno
Samples Notes: Opaque brick red colored capsule in containing yellow-orange powder

| Method     | Controls                         | Results | M.C. | Spec? | Lower lim |           | Upper lim Book-Data Analyses | Analyst    | Verifier |
|------------|----------------------------------|---------|------|-------|-----------|-----------|------------------------------|------------|----------|
| MESEUCO1NO | OBSIGUÇO1#6 CONTENT UNIFORMITY#1 | 98798   | ¥º   |       | 95.000    | - 115,000 | 143151 OCT 2001              | CAPPUCCE   | MACCAR   |
| _          | CONTENT UNIFORMITY#2             | 98.260  | ×    |       | 95,000    | - 115.000 | 1430M OCT 200M               | CAPPLICCIO | MACCAR   |
|            | CONTENT UNIFORMITY#3             | 98-926  | ×    |       | 95.000    | - 115,000 | 14310 CCT 2001               | CAPPUCCIO  | MACCAR   |
|            | CONTENT UNIFORMITY#4             | 45E-16  | ĸ    |       | 95,000    | - 115,000 | 145/01 OCT 2001              | CAPPUCCIO  | MACCAR   |
|            | CONTENT UNIFORMITY#5             | 100.192 | ×    |       | 85.000    | - 115.00Q | 145/04 OCT 2001              | CAPPLICEN  | MACCAR   |
|            | CONTENT UNIFORMITY#6             | 100,479 | ¥    |       | 95,000    | 115,000   | 143/01 OCT 2001              | CAPPUCCIO  | MACCAR   |
|            | ·CONTENT UNIFORMITY#7            | 101,163 | ×    |       | 95,000    | - 115,000 | 14301 OCT 2001               | CAPPUCCIO  | MACCAR   |
|            | CONTENT UNIFORMITY#8             | 56,637  | ¥    |       | 95.000    | - 115,000 | 143/01 OCT 2001              | CAPPUCCIO  | MACCAR   |
|            | CONTENT UNIFORMITY#9             | 746.98  | ¥    |       | 85.000 -  | 115,000   | 143/01 OCT 2001              | CAPPUCCIO  | MACCAR   |
|            | CONTENT UNIFORMITY#10            | 100.852 | ×    |       | 95,000    | 115,000   | 143/01 OCT 2001              | CAPPUCCIO  | NACCAR   |
|            | CONTENT UNIFORMITY               | 99.54   | *    |       | 85,000    | - 115,000 | 143/01/OCT 200H              | CAPPUCCIO  | MACCAR   |
|            | STANDARD DEVIATION               | 1,192   | .₹   |       |           |           | 143/01 OCT 2001              | CAFFUCCIO  | MACCAR   |
|            | C.V.                             | 1.2     | ×    |       |           |           | 142/01 OCT 2001              | CAPFUCCIO  | HACCAR   |
|            | CONTENT UNIFORMITY MAX           | 101.183 | ×    |       | 95,000    | - 115,000 | 143/0f OCT 2001              | CAPPLICCE  | NACCAR   |
| •          | CONTENT UNIFORMITY MIN           | 107.364 | *    |       | 85.000    | - 115,000 | 14201 OCT 2001               | CAPPLICCE  | ENCOME   |



Pharmacia & Upjohn

Integrated Analytical Bulletin

Laboratory Information Management System

Submission Str. 114906

Sample ID (Request No.): 20013158

User sample ID (tot): IE3G03

Sumple Date: D1/11/2001

Batch Manefecture Date: 17/09/2001 Makilly Shady: 85/603

Time Labet TIME ZERO Statege Condition: TIME ZERD Pack Type: BULK

Preparation purpose: CLINICAL SUPPLY + STABILITY

Condition: APPROVED

COMPLETE

Sample Type: SULYMORE (PNUZROBALAD) CAPSULE Stability Study Start Dale: 18/10/2001

Observations on the Method 083GDS01#\_12, component: DISSOLUTION

Opaque brick red colored capsule containing yellow-orange powder

Siekliky Protocol Number: 2001087 - Manufacturer: PD6Phirmacia Neviano

Samples Notes:

Dissolution after 15 minutes equal to: 66%

(min. 31.5%-max. 105.5%) Dissolution after 30 minutes equal to: 92.6%

(min. 88%-max. 101.9%) Dissolution after 45 minutes equal to: 97.5%

Observations on the Method 083GMC01#01, component: TOTAL AEROBIC BACTERIA

Total bacterial count < 20 Ufc/g

083GMC01#01, component: ENTEROBACTERIA Observations on the Method

Enterobactería < 10 Ufc/g

083GMC01#01, component: FUNGI Observations on the Method

Fungi (Yeasts + Molds) < 10 Ufc/g

Observations on the Method 083GSC01#00, component: IMPURITIES NOT LESS THAN 0.05%

Total correlated substances: 0.273%

Area % 뇶 999

0.064 0.062 0.107 0.107 \$ 5 5 5 5 5 5

(SU 11246)

083GTC18#01\_IR, component: (HPLC TITER) SAMPLE #3 Observations on the Method

NACCAR ፩ Printed 30-JAN-2002 10:55 AM

Page

5 6



Laboratory Information Management System

Sample ID (Request No.): 20013188 User sample ID (lot): 183663 Subretesion 14: 114906

Sumple Type: SUditass (PNUSSOS(GAD) CAPSULE 25646 Serryto Date : 01/11/2005

Babck Wanniecher Date: 17/00/2001 Steintly Study: 163003

Storage Condition: TIME ZERO These Labot: TIME ZENO Pack Typec BULK

Preparation purpose: CLINICAL SUPPLY + STABILITY

APPROVED

Status: COMPLETE

Stability Study Start Date: 16/10/2001

Statisticy Protocol Number: 2001017 - Manufacture: PDP harmacle Newford

Opaque brick red colored capsule containing yellow-orange powder Samples Notes:

Observations on the Method 083GTC18#01\_IR, component: (HPLC TITER) SAMPLE#3

Average titer determined based on nine different samples

Observations on the Method 083GTC18#01\_IR, component: AVERAGE TITER (HPLC)

Approved by: MACCARI Approval date: 26-104-2001

MACCAR 호 30-JAN-2002 10:55 AM Printed

Page